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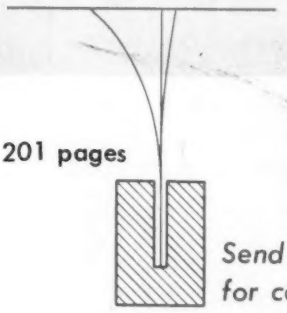

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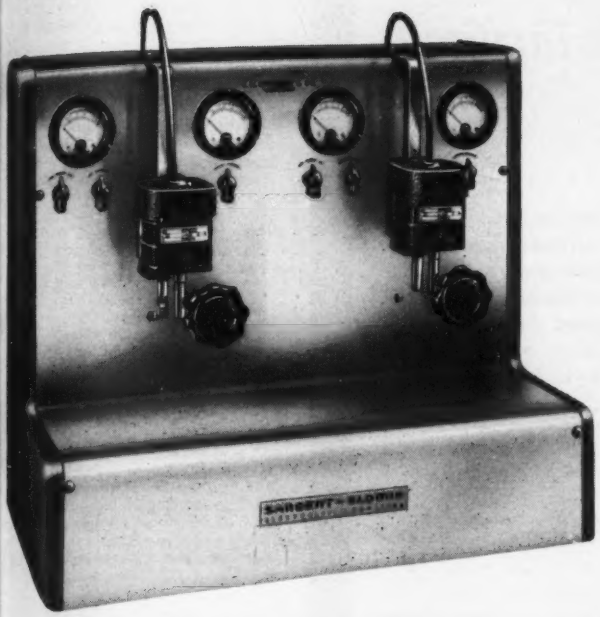

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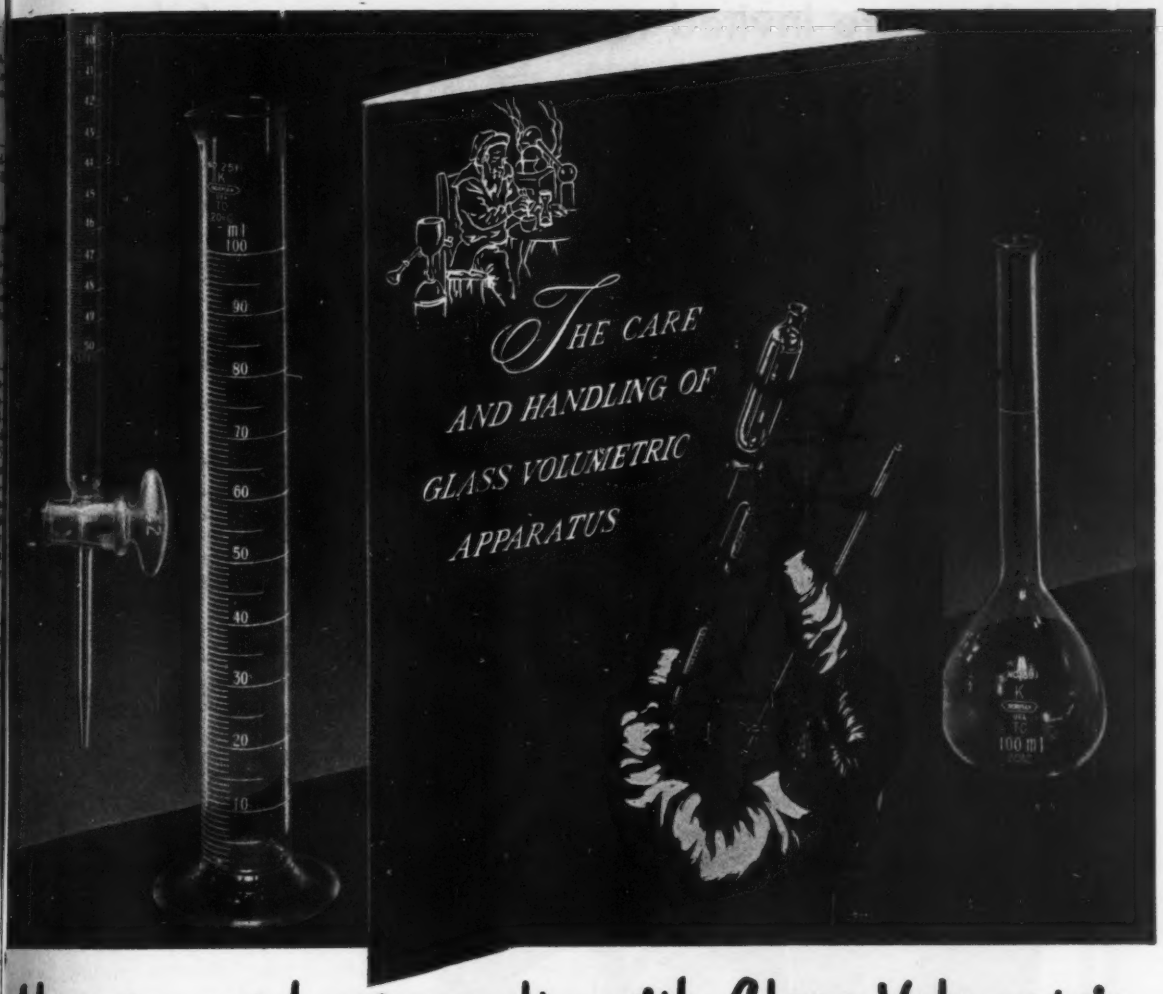

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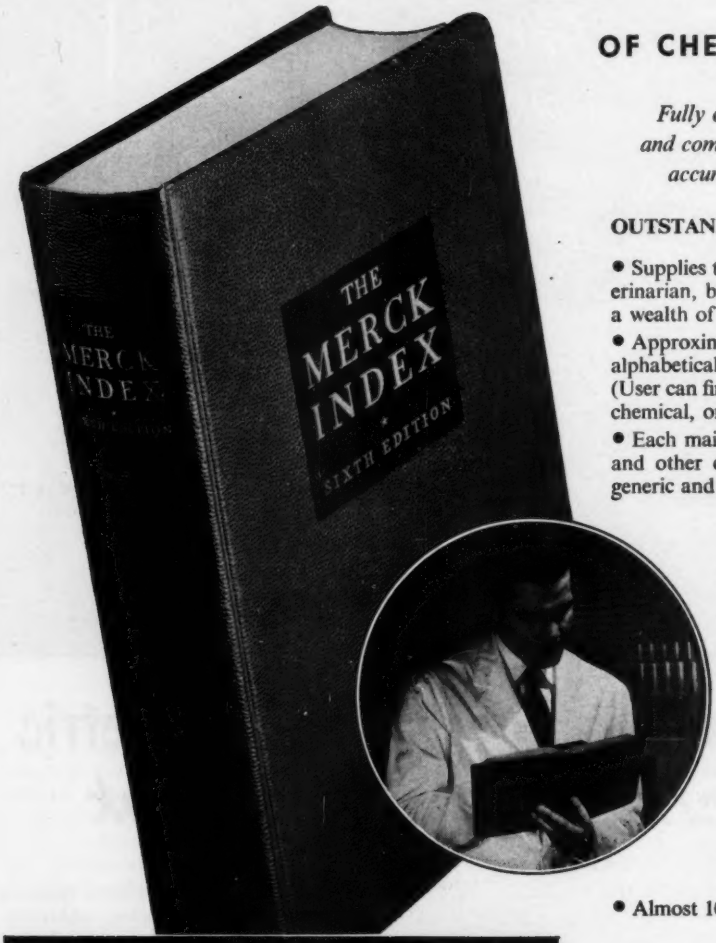

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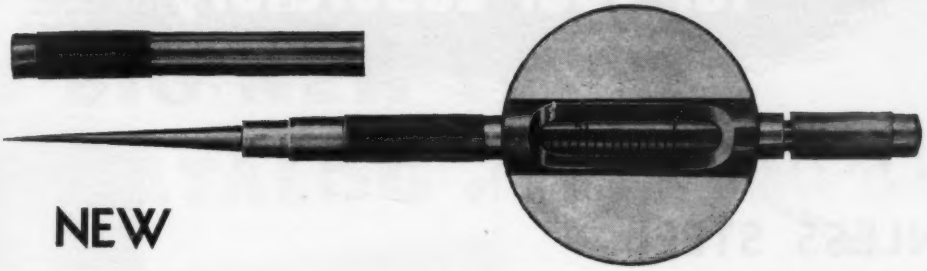

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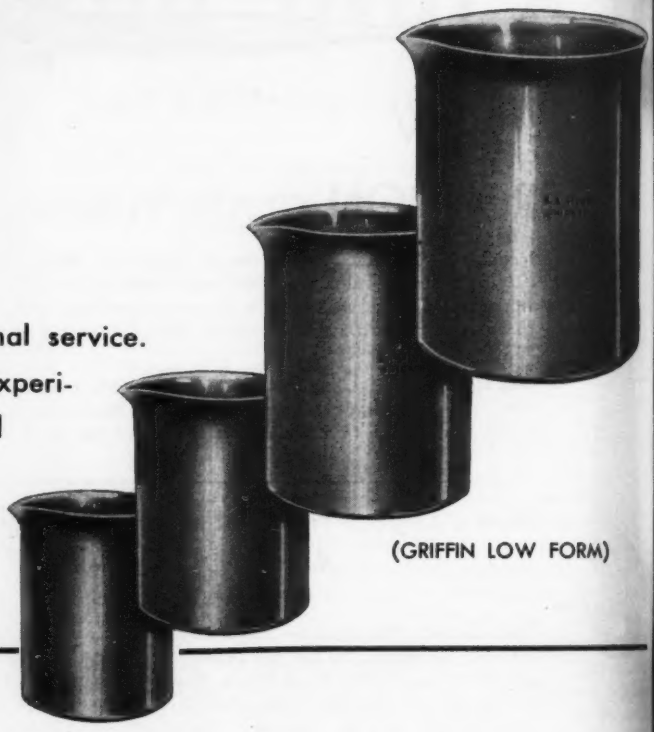

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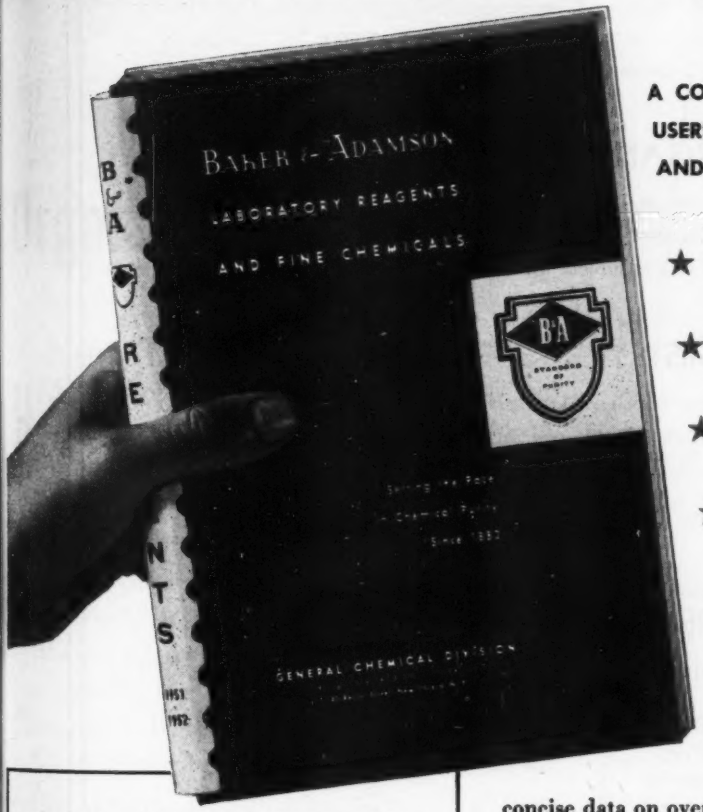

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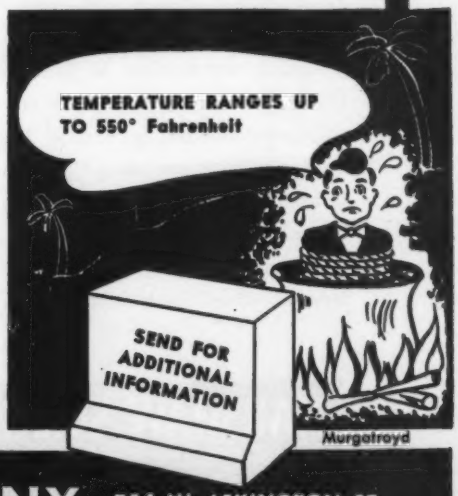

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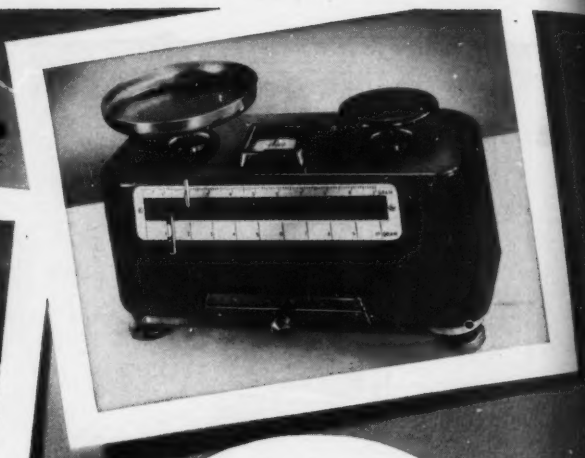

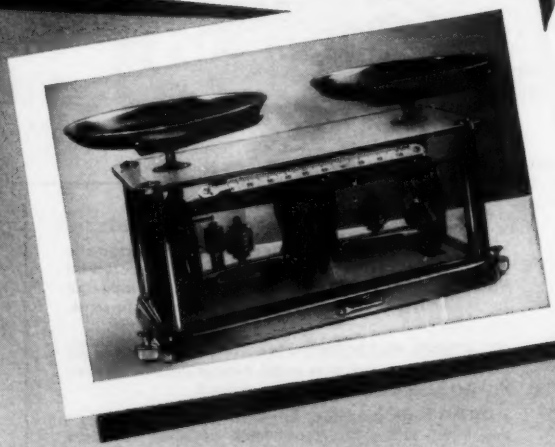

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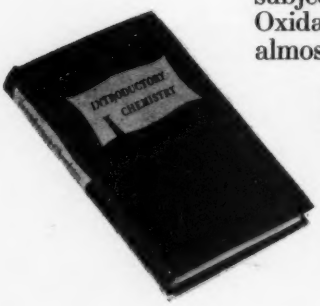

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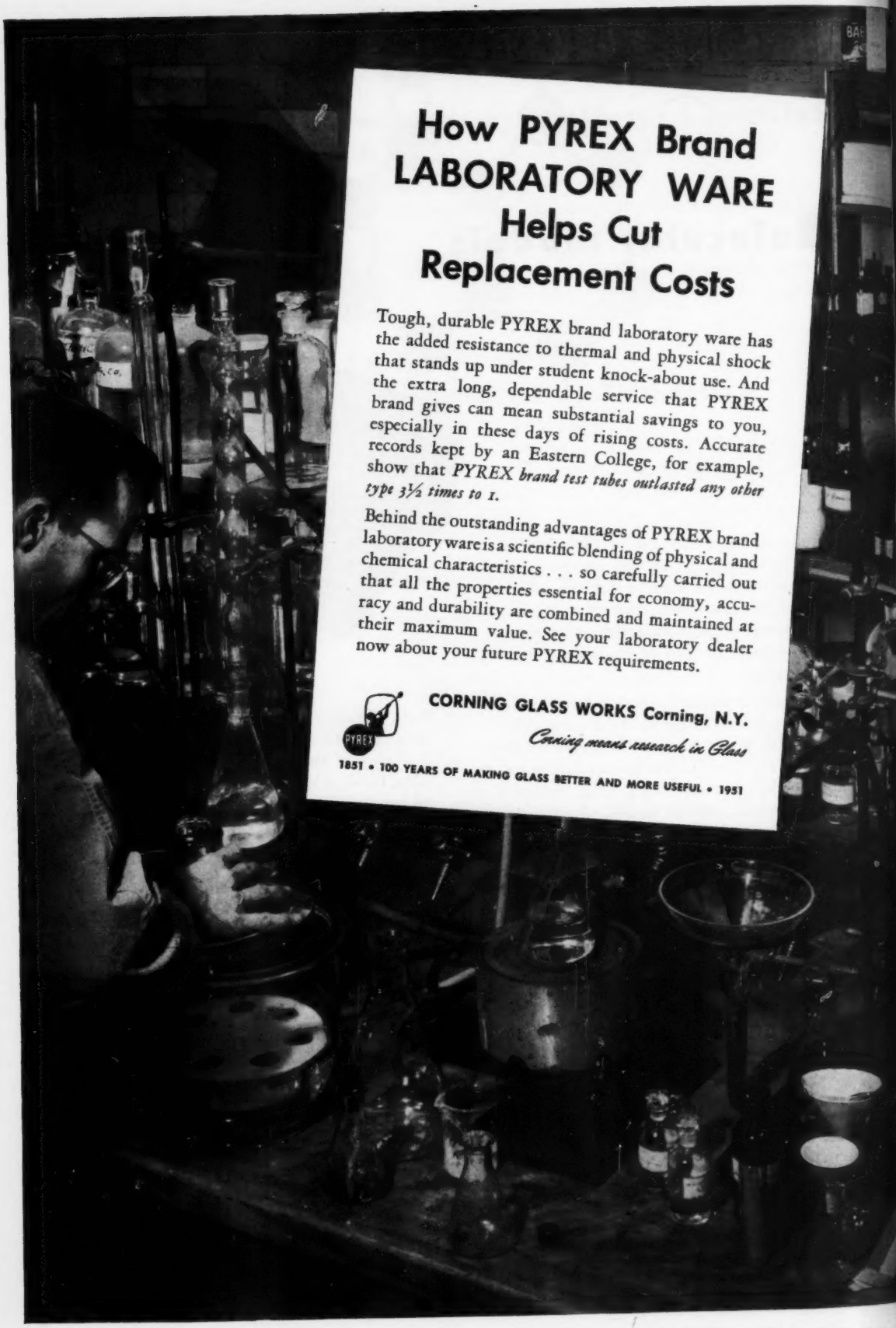
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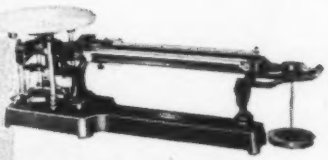

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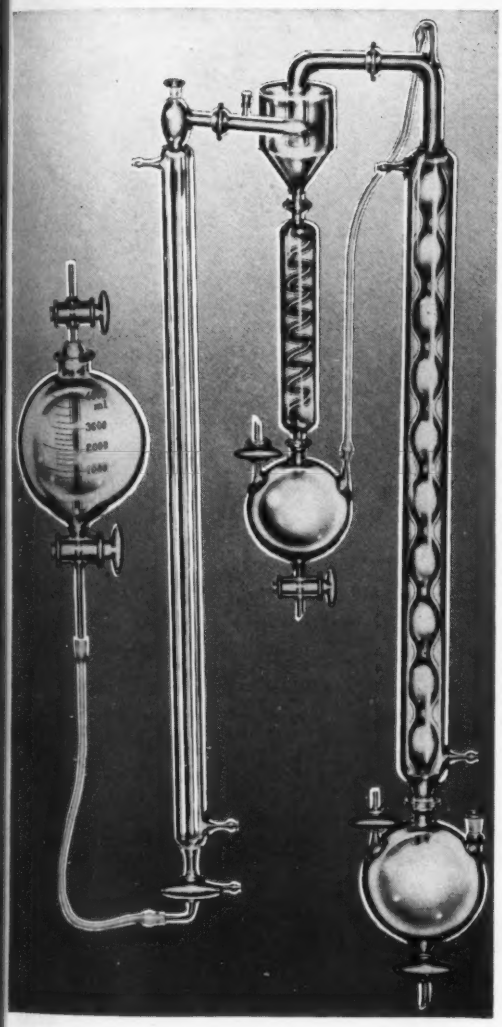

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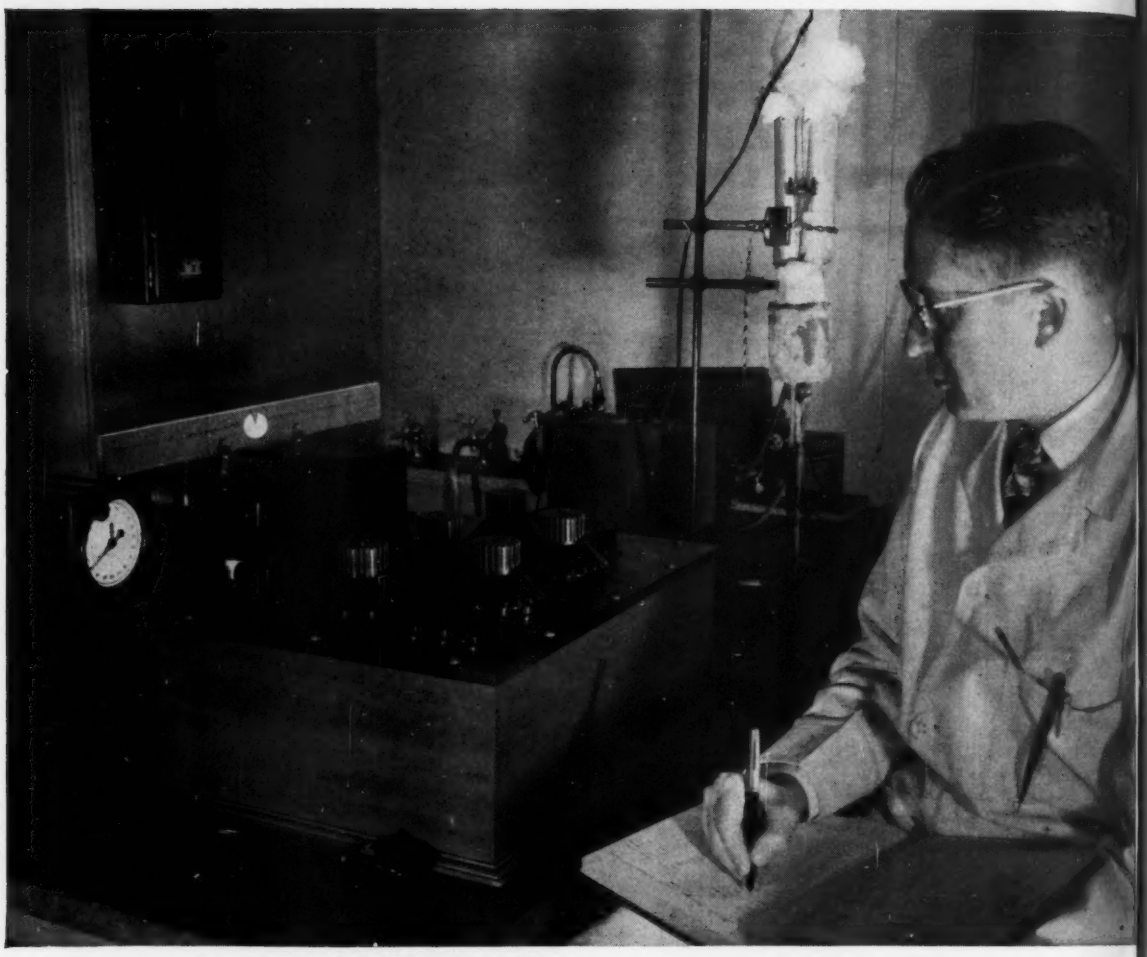

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(See page 575)

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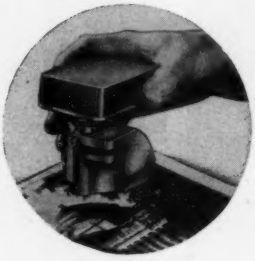

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Editor's Outlook

I HAVE always maintained that chemical education scarcely begins—and certainly does not end—with the school and classroom. Many agencies and institutions are involved in it beyond the college and university. Industry is concerned in it, in other ways than merely absorbing the product of the process.

For industry is, of course, the principal user of chemists and should therefore do its fair share in making them. Industrial training programs first come to mind. The new employee, usually fresh from the collegiate ivory tower, is seldom ready to jump into the industrial routine without a period of on-the-job training. Sometimes this is a hit-or-miss, trial-and-error process; occasionally it is a highly organized training program in which the new employees learn the real fundamentals and details of the jobs they are about to undertake. The academic institutions are beginning to depend upon such industrial training programs, recognizing that they should *educate* rather than *train*, and that if they can turn their product out with a good all-round preparation and with a sound knowledge of basic fundamentals they will best serve the interests of industry, for it will be manifestly impossible to fit every man completely for the job ahead.

Sometimes these industrial training programs are conceived in a very long-time view, even beyond the particular industry concerned—such as the Seagram's "fellowship" program, in which young men are brought even from abroad to learn (by *doing*) some of the details of a particular industry.

The responsibility of industry to the teachers of science has been tangibly recognized by such means as the General Electric Science Fellowships, with their inspiring summer sessions at the Company's plant; also by the extended plant trips and "teachers' institutes" which several industries have established.

Industries find it "good public relations" to be cooperative with the schools and make available literature, demonstration kits, and teaching gadgets of all sorts. Welcome as all this help and attention are, it is highly colored by the advertising motive and probably pays for itself in the long run. The same may be true of industrial fellowships, of long standing in universities and technical schools.

But the debt is far from paid, and industry may expect to be called upon for further contributions to technical education. These are profitable years, for industry but not for the colleges and universities. Many an institution is seriously wondering where its next year's income is going to come from—especially the smaller arts colleges. But these same small colleges are turning out more than their share of chemists, some of them, and are probably getting very little of the direct or indirect help that larger institutions get from industry. We do not dare let our small colleges run down. What are we going to do about them? Industry can help us find the answer.

THE MECHANISMS OF THE REACTIONS OF ALIPHATIC HYDROCARBONS[1]

LOUIS SCHMERLING
Universal Oil Products Company, Riverside, Illinois

In general, the large variety of reactions of the aliphatic hydrocarbons can be divided into two classes, (1) those which take place in the presence of acid-type catalysts, such as the Friedel-Crafts metal halides, on the one hand, and acids such as sulfuric acid and hydrogen fluoride, on the other, and (2) those which occur thermally or are induced by peroxides. It is the object of the present paper to show that the reactions of the first class can best be explained as occurring by way of a carbonium ion, usually chain, mechanism, while reactions of the second class involve free radicals. It will be shown that by making a few plausible assumptions and by setting up certain principles, now generally accepted, of the behavior of carbonium ions and free radicals, the difference between the two classes of reactions can be readily understood.

Substantially all of the hydrocarbon reactions catalyzed by acid-type catalysts can be explained as involving (1) addition of a carbonium ion to a molecule via a pair of pi electrons to yield a higher molecular weight carbonium ion, (2) decomposition of a carbonium ion to another ion (*e. g.*, a smaller carbonium ion or a proton) and an unsaturated compound, (3) isomerization of a carbonium ion via migration of hydrogen or alkyl together with a pair of electrons, or (4) a combination of these. The final carbonium ion is converted to the reaction product either by eliminating a proton or other cation or by abstracting a hydride ion from a molecule of reactant.

In an analogous manner a free radical, the usual intermediate in thermal and peroxide-induced reactions, can (1) add to an unsaturated bond to form a higher molecular weight free radical, (2) decompose to yield a smaller radical and an unsaturated compound, (3) abstract a hydrogen atom or other atom from a molecule to yield a new molecule and a new free radical, and (4) lose an atom, usually hydrogen, to form an unsaturated compound. Unlike carbonium ions, free radicals rarely isomerize. Free alkyl radicals readily undergo disproportionation to yield a paraffin and an olefin.

It is obviously beyond the scope of this paper to give a comprehensive discussion of all the species of reactions which have been studied. Instead, typical examples will be given illustrating the basic principles involved.

FORMATION OF CARBONIUM IONS

From Olefins. Carbonium ions can be formed in a number of ways, some of which will be reviewed here. With acid catalysts (*e. g.*, sulfuric acid) an olefin is converted to a carbonium ion by addition of the hydrogen ion from the acid to the extra electron pair in the double bond (the pi electrons):

$$H_2C::C(\ddot{C}H_3):CH_3 + H^+OSO_3H^- \rightleftharpoons H_2C:\overset{+}{C}(\ddot{C}H_3):CH_3 + OSO_3H^-$$

With halide catalysts of the Friedel-Crafts type (*e. g.*, aluminum chloride or boron fluoride), in the presence of hydrogen halide, the carbonium ion is formed by the addition of the proton from the promoter to the pi electrons of the olefin:

$$H_2C::CH_2 + HCl + AlCl_3 \rightleftharpoons H_2C:CH_2^+ + AlCl_4^-$$

Recent work has indicated that while hydrogen aluminum tetrachloride ($HAlCl_4$) is nonexistent, interaction of aluminum chloride and hydrogen chloride does occur in the presence of substances (such as benzene and, presumably, olefins) to which basic properties may be ascribed (*3*). In other words, while hydrogen aluminum tetrachloride is an unstable acid, its esters are fairly stable. Additional evidence, in support of the hypothesis that such esters are intermediates in the Friedel-Crafts reaction, is found (*8*) in the fact that exchange of radioactive chlorine atoms for ordinary chlorine atoms occurs when *t*-butyl chloride is treated with benzene in the presence of aluminum chloride containing radioactive chlorine atoms; the hydrogen chloride which is evolved is radioactive.

$$(CH_3)_3CCl + AlCl_3^* \rightleftharpoons (CH_3)_3C^+[AlCl_4^*]^-$$

$$(CH_3)_3C^+[AlCl_4^*]^- + C_6H_6 \rightleftharpoons (CH_3)_3CC_6H_5 + [HAlCl_4^*]$$

$$[HAlCl_4^*] \rightarrow HCl^* + AlCl_2^*$$

(Cl* is chlorine, part of which is radioactive)

The Friedel-Crafts catalysts are electron acceptors, and, in the absence of hydrogen halide promoter, the active complex is presumably formed by the addition of the catalyst to the olefin (*13*):

$$\begin{matrix} :\ddot{C}l: & H & H \\ :\ddot{C}l:\ddot{A}l & + & \ddot{C}::\ddot{C} \\ :\ddot{C}l: & H & H \end{matrix} \rightarrow \begin{matrix} :\ddot{C}l:H\ H \\ :Cl:\ddot{A}l:\ddot{C}:\ddot{C}^+ \\ :\ddot{C}l:H\ H \end{matrix}$$

This complex, while it contains an electron-deficient (hence, positive) carbon atom, is in itself electronically neutral; the product of the addition of a proton to an olefin contains a similar carbon atom but is in itself electrically positive. It has been suggested (*46*) that this difference is related to the fact that metal halide catalysts tend to yield much higher polymers than do

[1] Presented before the Division of Petroleum Chemistry at the 119th Meeting of the American Chemical Society, Cleveland, April 10, 1951.

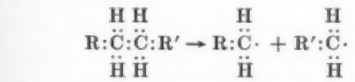
the acid (proton) catalysts. However, it may be significant that water or another promoter is usually necessary for polymerization in the presence of the metal halide if hydrogen halide is not present.

From Paraffins. As will be shown below in the discussion of the hydrogen-halogen exchange reaction, paraffins are converted to alkyl carbonium ions by loss of a hydride ion to a carbonium ion.

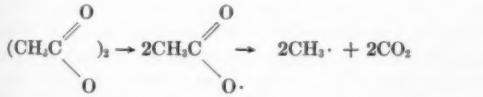
It should be remembered that while carbonium ions are often written (for reasons of convenience) as if standing alone (*e. g.*, $(CH_3)_3C^+$), the negative portion of the ionic pair (*e. g.*, $AlCl_4^-$ or OSO_3H^-) plays an essential, if lesser, role. Also, while it has become customary to speak of the reactions of the carbonium ions as involving migration (or elimination) of an atom or group of atoms (*e. g.*, hydrogen or methyl) with (or, in the case of elimination, without) the attached electron pair, a useful and perhaps more accurate viewpoint is that of Whitmore which considers the changes as involving migration or elimination of an electron pair with or without the attached atom or group of atoms (*44*).

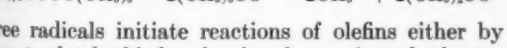
FORMATION OF FREE RADICALS

Free radicals are formed from paraffins and olefins at high temperatures by scission of a carbon-carbon bond:



Free radicals are also formed by the thermal decomposition of peroxides at comparatively low temperatures. This is a widely used method for obtaining the free radicals necessary for inducing a large variety of reactions.

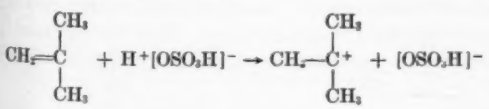


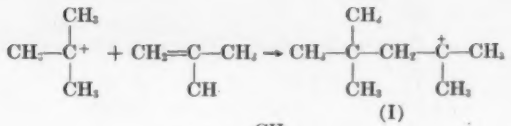


Free radicals initiate reactions of olefins either by adding to the double bond or by abstracting a hydrogen atom; they initiate paraffin reactions by abstracting hydrogen atoms.

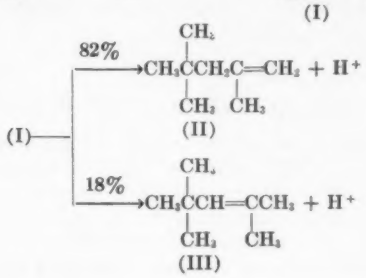
POLYMERIZATION OF OLEFINS

Catalytic Dimerization of Isoolefins. The dimeric product obtained by the polymerization of isobutylene in the presence of acidic catalysts consists of a mixture of trimethylpentenes. With dilute sulfuric acid, 2,4,4-trimethyl-1-pentene (II) and 2,4,4-trimethyl-2-pentene (III) are produced in the ratio of about four to one (*22*). Their formation is best explained by the well-known Whitmore mechanism (*43*):





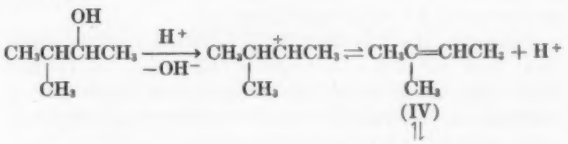
(I)



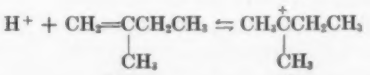
(II) (III)

The polymerization involves the condensation of isobutylene with a *t*-butyl carbonium ion (formed by addition of the catalyst to isobutylene) to yield a tertiary octyl ion (I). Loss of a proton yields a trimethylpentene and regenerates the catalyst. The 1-pentene derivative is formed in larger amount because elimination of one of the six protons on the two primary carbon atoms adjacent to the positively charged carbon atom takes place more readily than does loss of one of the two protons on the methylene carbon atom in the neopentyl group, a group which always loses protons with comparative difficulty.

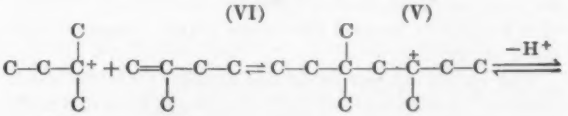
The products obtained when 3-methyl-2-butanol is treated with 75 per cent sulfuric acid at 90°C. may be explained in similar manner. Whitmore and Mosher (*47*) showed that the products include 2-methyl-2-butene (IV), 1 per cent; 3-methyl-2-pentene (X), 3 per cent; methyl isopropyl ketone, 1 per cent; diisobutylenes, 1 per cent; 2,3,4,4-tetramethyl-1-pentene, 2 per cent; other nonenes, 1 per cent; 3,4,5,5-tetramethyl-2-hexene (XI), 45 per cent; and 3,5,5-trimethyl-2-heptene (VII), 35 per cent. Oxidation of the original alcohol accounts for the presence of the methyl isopropyl ketone; sulfur dioxide was also a product of the reaction. Formation of the other products is explained as follows:



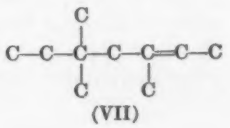
(IV)



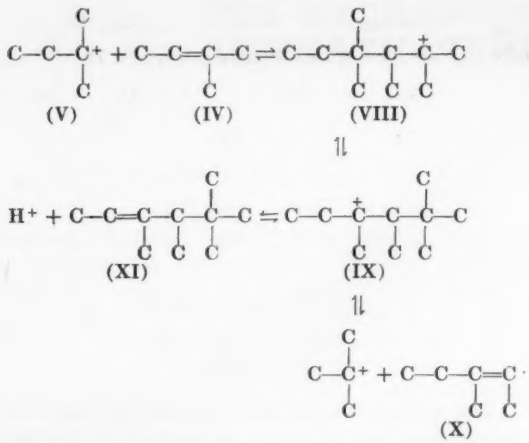
(VI) (V)



(V) (VI)



(VII)



Formation of 3-methyl-2-pentene and *t*-butyl carbonium ion (hence, isobutylene) from the decyl ion (IX) illustrates an important reaction of carbonium ions, namely scission at the bond in beta-position to the positively charged carbon atom. Such beta-scission takes place particularly readily in carbonium ions containing the grouping $R_3C—C—C^+$; it is the reverse of polymerization. It involves the shift of an electron pair to the adjacent deficient carbon atom without the attached alkyl group.

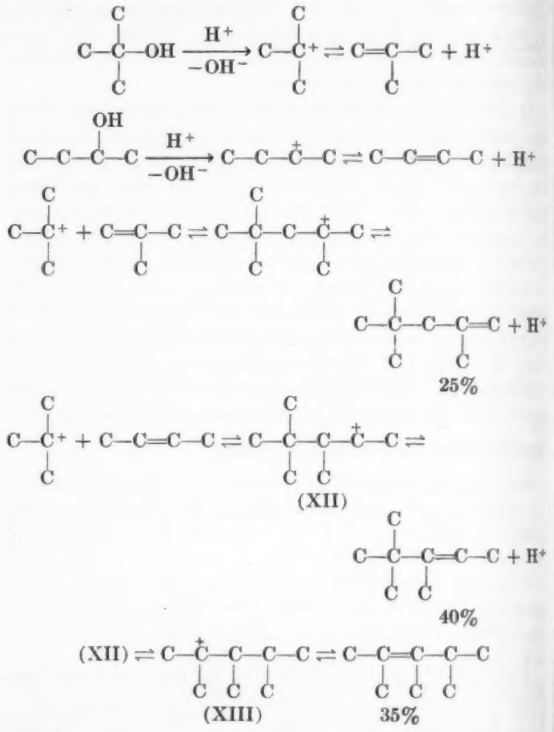
The isobutylene formed by the scission of (IX) polymerizes to yield diisobutylene and copolymerizes with 2-methyl-2-butene (IV) and other pentenes to yield, respectively, 2,3,4,4-tetramethyl-1-pentene and other nonenes.

Production of 3,5,5-trimethyl-2-heptene rather than the 3-heptene isomer is a further illustration of the relative difficulty of loss of a proton from the neopentyl system. This was confirmed experimentally by carrying out the dehydration of the corresponding carbinol under the same conditions that were used in the polymerization. Treating 3,5,5-trimethyl-3-heptanol at 80°C. with 75 per cent sulfuric acid resulted in the formation of 20 parts of the 3,5,5-trimethyl-2-heptene to only one of 3,5,5-trimethyl-3-heptene.

Similarly, the fact that 3,4,5,5-tetramethyl-2-hexene (XI) and not 3,4,5,5-tetramethyl-3-hexene is formed from (IX) again shows the comparatively slow rate of elimination of a proton from a neopentyl carbon atom even when it is a tertiary carbon atom.

It will be noted that the formation of (XI) as written involves a 1,3-shift of a methyl group. Whether such a shift occurs or whether it actually involves a succession of 1,2-shifts is beyond the scope of the present paper (*cf. 4, 5, 24, 48*).

The copolymerization of isobutylene with the *n*-butylenes (as investigated (*45*) by the addition of *t*-butyl alcohol to an equimolecular quantity of *s*-butyl alcohol in the presence of 75 per cent sulfuric acid at 64°) results in a 78 per cent yield of octene, consisting of 2,4,4-trimethylpentenes (25 per cent), 3,4,4-trimethyl-2-pentene (40 per cent), and 2,3,4-trimethyl-2-pentene (35 per cent). The formation of all these products may again be explained on the basis of the addition of a *t*-butyl ion to an olefin:



The octenes appear to be produced via the addition of the *t*-butyl ion to isobutylene and 2-butene to the extent of 25 and 75 per cent, respectively, about half of the products in the latter case being formed by rearrangement. 1-Butene is not formed as an intermediate because loss of a proton occurs more readily from the methylene group than from the methyl group. None of the products which would be formed by the addition of the *s*-butyl carbonium ion to the 2-butene or to isobutylene were obtained. The rate of addition of the *s*-butyl ion to the double bond is apparently too slow; this finds analogy in the comparative ease of addition of *t*-butyl chloride and *s*-butyl chloride to ethylene in the presence of Friedel-Crafts type catalysts (*30*).

Macropolymerization of Isobutylene. The polymerization of isobutylene at temperatures below about −70°C. in the presence of Friedel-Crafts catalysts (particularly boron fluoride, aluminum chloride, and titanium chloride) yields a high molecular weight polymer having elastic properties.

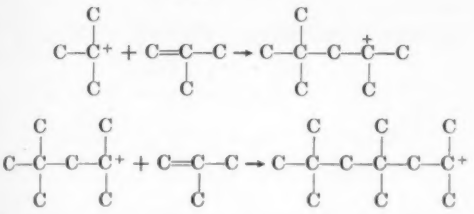
No reaction occurs when carefully purified isobutylene is contacted with pure boron fluoride at −80°C. (*6*). There is instantaneous reaction when water or *t*-butyl alcohol is added to the mixture. The boron fluoride is consumed in quantities approximately equivalent to the

mols of third substance added. The polymer is apparently formed by head-to-tail polymerization; the polymer molecules presumably usually contain one terminal double bond.

The following mechanism (*7*) explains the experimental observations:

$$BF_3 + HY \rightarrow F_3BYH \qquad (Y = OH, OR, \text{etc.})$$

$$F_3BYH + CH_2{=}C(CH_3){-}CH_3 \rightarrow F_3BY^- + (CH_3)_3C^+$$



The chain continues to grow until terminated by one of the following reactions in which M represents monomer (C_4H_8) and n is the number of molecules reacted.

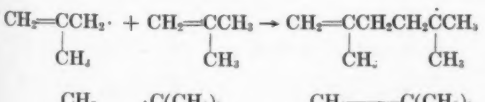
$$M_nH^+ + F_3BY^- \rightarrow M_n + F_3BYH$$

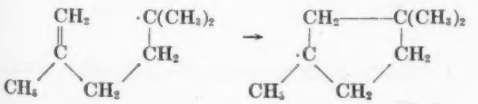
$$M_nH^+ + F_3BY^- \rightarrow M_nHY + BF_3$$

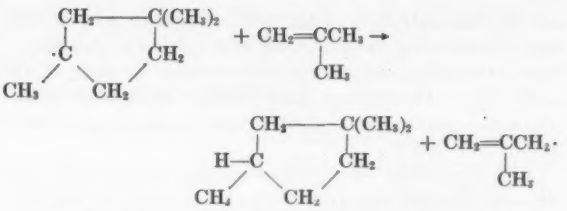
$$M_nH^+ + M \rightarrow M_n + MH^+$$

Thermal Dimerization of Isobutylene. The thermal polymerization of isobutylene (at 370–460°C. and 540–5350 p. s. i.) is of particular interest because it yields 1,1,3-trimethylcyclopentane rather than 2,4,4-trimethyl-1- and 2-pentene, the products of the catalytic polymerization (*23*). For example, the cyclic dimer comprised about 46 per cent of the total liquid product when the reaction was carried out at 400° and 540 p. s. i. A number of mechanisms were proposed to explain its formation but none of these seems as plausible as the following chain mechanism which is based on the principles enumerated above. It postulates an intramolecular alkylation step which is directly analogous to that which has been proposed for intermolecular thermal alkylation, namely addition of an alkyl radical to the double bond of the alkene (see subsequent discussion). The method of formation of the chain initiator, R′·, is not critical since it merely starts the first cycle of the chain reaction; it may be formed by decomposition of the isobutylene or by oxygen present in the reaction mixture or by other means.

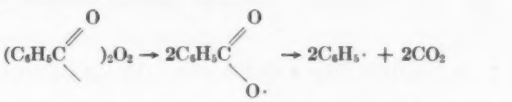
$$R'\cdot + CH_2{=}C(CH_3){-}CH_3 \rightarrow R'H + CH_2{=}C(CH_3){-}CH_2\cdot$$







Peroxide-Induced Polymerization of Ethylene. The polymerization of ethylene under high pressure in the presence of oxygen or peroxide yields very high molecular weight polymers. For example, polymerization at 110–120°C. and 200–300 atm. pressure in the presence of a methanol solution of benzoyl peroxide yields a wax, melting at 105–110°C., having a molecular weight of 2000–3000 and containing 0.7–1.3 per cent of oxygen. A free-radical chain reaction mechanism (*9, 27, 40*) is undoubtedly involved, the chain being initiated by benzoyloxy or phenyl radicals:



$$C_6H_5COO\cdot + CH_3OH \rightarrow C_6H_5COOH + CH_3O\cdot$$

$$C_6H_5\cdot + CH_3OH \rightarrow C_6H_6 + CH_3O\cdot$$

The chain is propagated by addition of intermediate radicals to ethylene molecules.

$$R\cdot + CH_2{=}CH_2 \rightarrow RCH_2CH_2\cdot$$

$$RCH_2CH_2\cdot + (m+1)CH_2{=}CH_2 \rightarrow RCH_2CH_2(CH_2CH_2)_mCH_2CH_2\cdot$$

The nature of R· (which may be methoxy, phenyl, or benzoyloxy) is relatively unimportant since it serves only to initiate the chain, which is continued by methoxy radicals formed by chain transfer.

Chain transfer involving the methanol solvent terminates the growing chain and at the same time yields a methoxy radical which starts a new cycle. The methoxy radical accounts for the presence of oxygen in the wax.

$$RCH_2CH_2(CH_2CH_2)_mCH_2CH_2\cdot + CH_3OH \rightarrow RCH_2CH_2(CH_2CH_2)_mCH_2CH_3 + CH_3O\cdot$$

The chain is terminated by coupling or disproportionation reactions involving two radicals (not necessarily alike):

$$2R(CH_2CH_2)_nCH_2CH_2\cdot \rightarrow R(CH_2CH_2)_n(CH_2)_4(CH_2CH_2)_nR$$

$$2R(CH_2CH_2)_nCH_2CH_2\cdot \rightarrow R(CH_2CH_2)_nCH{=}CH_2 + R(CH_2CH_2)_nCH_2CH_3$$

Conjunct Polymerization. The term conjunct polymerization has been given to that type of catalytic polymerization which results in the formation not only of higher molecular weight olefins but also of saturated hydrocarbons (*18*). With some catalysts, for example with aluminum chloride, true polymerization of the olefin takes place only under special conditions (*12, 15*). With sulfuric acid the type of polymerization depends

on the concentration of the acid; conjunct polymerization occurs with concentrated acid (above about 90 per cent) while true polymerization occurs with more dilute acid (*17*). Phosphoric acid yields conjunct polymers when the polymerization occurs at temperatures above about 250–300°C. (*16*).

Polymerization of ethylene in the presence of aluminum chloride may be cited as an example of conjunct polymerization. While no polymerization at all occurs even at superatmospheric pressures and 10–50° when pure aluminum chloride is contacted with pure ethylene, polymerization occurs quite readily in the presence of hydrogen chloride or traces of impurities. The product consists of a water-white, chiefly paraffinic, upper layer and a viscous, dark red-brown lower layer consisting of addition compounds of aluminum chloride and highly unsaturated hydrocarbons. The reaction can be explained in terms of the carbonium ion mechanism by assuming that the olefin which would be the product of true polymerization undergoes further reaction as follows (*1*):

$$RCH_2CH_2CH{=}CHR' + HCl + AlCl_3 \rightleftharpoons RCH_2CH_2\overset{+}{C}HCH_2R' + AlCl_4^-$$

$$RCH_2CH_2\overset{+}{C}HCH_2R' + RCH_2CH_2CH{=}CHR' \rightleftharpoons RCH_2CH_2CH_2CH_2R' + RCH_2\overset{+}{C}HCH{=}CHR' \quad \text{(XIV)}$$

$$RCH_2\overset{+}{C}HCH{=}CHR' + AlCl_4^- \rightleftharpoons RCH{=}CHCH{=}CHR' + HCl + AlCl_3$$

The final products are thus paraffins and polyolefins. The latter are present in the catalyst layer.

HYDROGEN-HALOGEN EXCHANGE

Metal Halide-catalyzed Reaction. Evidence that a carbonium ion can abstract a hydride ion as indicated in the above mechanism of conjunct polymerization was obtained in an investigation of the reaction of alkyl halides with isoparaffins (*1*). It was shown that hydrogen-halogen exchange occurs when, for example, a mixture of isopentane and *t*-butyl chloride is treated with aluminum chloride for as short a time as 0.001 sec. The mechanism of this exchange appears to involve the removal of a hydride ion from isopentane by the *t*-butyl carbonium ion formed from the alkyl chloride:

$$(CH_3)_3CCl + AlCl_3 \rightleftharpoons (CH_3)_3C^+AlCl_4^-$$

$$(CH_3)_3C^+ + C_2H_5(CH_3)_2CH \rightleftharpoons (CH_3)_3CH + C_2H_5(CH_3)_2C^+$$

$$C_2H_5(CH_3)_2C^+ + AlCl_4^- \rightleftharpoons C_2H_5(CH_3)_2CCl + AlCl_3$$

Since tertiary alkyl carbonium ions are more stable than secondary alkyl ions, isoparaffins undergo the hydrogen-halogen exchange reaction more easily than do *n*-paraffins.

Ions of the allyl type (*e. g.*, XIV) are stabilized by resonance and are therefore readily formed from olefins by abstraction of a hydride ion in allylic position.

A somewhat similar reaction occurs with polyhaloalkanes. Carbon tetrachloride is converted to chloroform when treated with saturated hydrocarbons and aluminum chloride (*19*).

Peroxide-induced Reaction. Hydrogen-halogen exchange also occurs when a polyhaloalkane, such as carbon tetrachloride, is treated with a paraffin in the presence of a free radical forming substance such as a peroxide (*38*). For example, the reaction of isobutane with carbon tetrachloride in the presence of di-*t*-butyl peroxide at 130–140°C. results in practically quantitative yield of *t*-butyl chloride and chloroform. It presumably occurs by way of a free radical chain mechanism:

$$(t\text{-}C_4H_9O)_2 \rightarrow 2t\text{-}C_4H_9O\cdot$$

$$t\text{-}C_4H_9O\cdot + CH_3CHCH_3 \rightarrow CH_3\dot{C}CH_3 + t\text{-}C_4H_9OH$$

$$CH_3\dot{C}CH_3 + CCl_4 \rightarrow Cl_3C\text{-}\overset{Cl}{\overset{|}{C}}\text{-}C + Cl_3C\cdot$$

$$Cl_3C\cdot + CH_3CHCH_3 \rightarrow Cl_3CH + CH_3\dot{C}CH_3$$

It is thus apparent that paraffinic hydrocarbons are capable of donating hydrogen atoms or hydride ions in reactions promoted by free radicals or carbonium ions, respectively. It is important to note that not only isoparaffins, but also normal paraffins containing at least three carbon atoms, are reactive in the free radical-induced hydrogen-halogen exchange. In the metal halide-catalyzed reaction, on the other hand, only isoparaffins react readily. The significance of this observation will become apparent in the discussion of the alkylation of paraffins.

CONDENSATION OF HALOALKANES WITH ALKENES

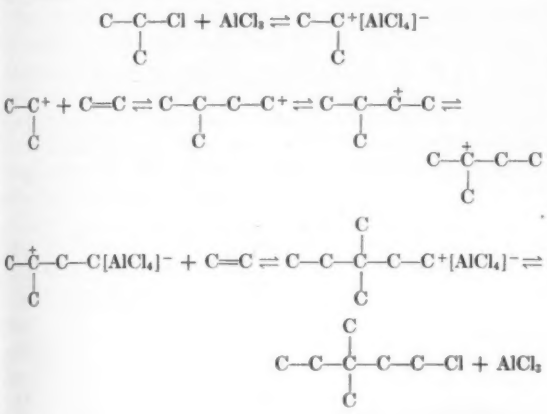
Metal Halide-catalyzed Reaction. Alkyl halides containing at least three carbon atoms can be condensed with olefins (*30, 35*) or haloolefins (*32, 36*) in the presence of catalysts of the Friedel-Crafts type to yield higher molecular weight haloalkanes. The primary reaction is the addition of the alkyl group and the halogen to the double bond of the alkene, the halogen adding to the carbon atom that holds the smaller number of hydrogen atoms. The reaction mechanism may best be expressed in terms of carbonium ion theory. Thus, the condensation of *t*-butyl chloride with ethylene in the presence of aluminum chloride yields 1-chloro-3,3-dimethylbutane:

$$C\text{-}\overset{C}{\overset{|}{C}}\text{-}Cl + AlCl_3 \rightleftharpoons C\text{-}\overset{C}{\overset{|}{C}}{}^+AlCl_4^-$$

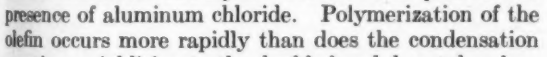
$$C\text{-}\overset{C}{\overset{|}{C}}{}^+AlCl_4^- + C{=}C \rightleftharpoons C\text{-}\overset{C}{\overset{|}{C}}\text{-}C\text{-}C^+AlCl_4^-$$

$$C\text{-}\overset{C}{\overset{|}{C}}\text{-}C\text{-}C^+AlCl_4^- \rightleftharpoons C\text{-}\overset{C}{\overset{|}{C}}\text{-}C\text{-}C\text{-}Cl + AlCl_3$$

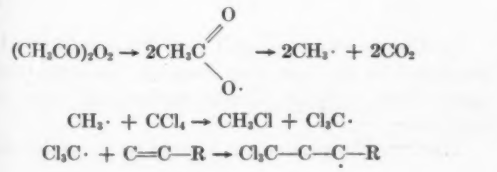
In similar manner, the condensation of isopropyl chloride with ethylene yields 1-chloro-3,3-dimethylpentane, *t*-pentyl chloride being an intermediate in the reaction. The primary reaction product, isopentyl chloride, contains a tertiary carbon atom and isomerization of the intermediate primary carbonium ion to the tertiary carbonium ion readily occurs. Tertiary carbonium ions add more readily to olefins than do secondary carbonium ions; hence, the *t*-pentyl ion adds more rapidly than does the isopropyl ion from the initial isopropyl chloride and the final product is therefore the 1-chloro-3,3-dimethylpentane (which contains no tertiary carbon atom).

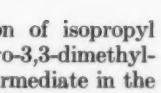


Attempts to condense polyhaloalkanes containing two or more halogen atoms on a single carbon with olefins in the presence of Friedel-Crafts catalysts have been unsuccessful (*28*). For example, addition of carbon tetrachloride to ethylene does not take place in the presence of aluminum chloride. Polymerization of the olefin occurs more rapidly than does the condensation reaction. Addition to the double bond does take place if a polychloroethylene is used instead of ethylene (*28*); the reaction of carbon tetrachloride with trichloroethylene yields *sym*-heptachloropropane, trichloromethylaluminum tetrachloride being formed as intermediate.



Peroxide-induced Reaction. On the other hand, condensation of polyhaloalkanes with olefins will occur in the presence of decomposing peroxide. For example, acetyl peroxide induces the reaction of carbon tetrachloride with 1-octene; 1,1,1,3-tetrachlorononane is formed (*20*). A free radical chain mechanism is involved, the trichloromethyl radical acting as chain carrier:



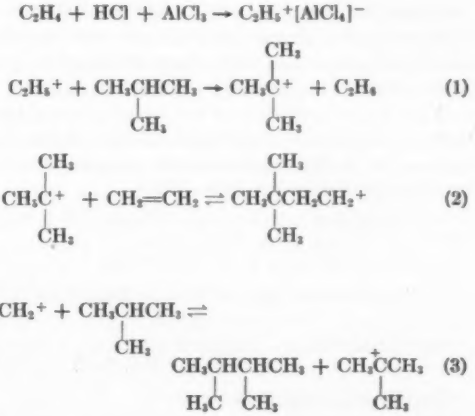


ALKYLATION OF PARAFFINS

It is well established that different products are obtained by the thermal alkylation of isobutane than by the catalytic alkylation. The differences are inherently explained by the principles already discussed for the behavior of free radicals and carbonium ions. No new concepts need be introduced.

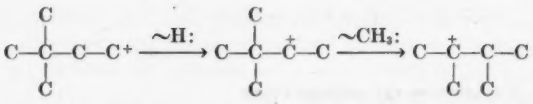
Catalytic Alkylation. The major product of the reaction of isobutane with ethylene in the presence of aluminum chloride or other active catalysts of the Friedel-Crafts type is 2,3-dimethylbutane. Isopentane and ethane are obtained as by-products. Similarly, the principal product of the reaction of isobutane with propylene in the presence of Friedel-Crafts catalysts or of acids is 2,3-dimethylpentane and 2,4-dimethylpentane; propane and 2,2,4-trimethylpentane are by-products of the reaction. A chain reaction involving carbonium ions explains all the products of the reactions (*1*, *29*).

Thus, for ethylene:

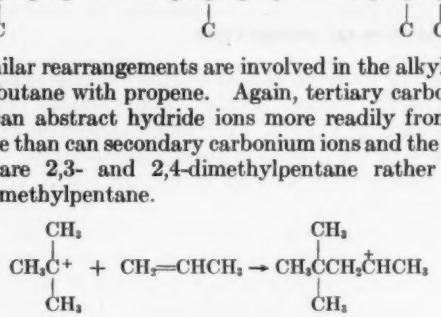


It will be noted that ethane is formed in the chain initiating step (equation (1)) only; the amount is therefore small. The reaction of equation (1) is obviously the hydrogen-halogen exchange reaction described above; it occurs much more readily with isoparaffins than with *n*-paraffins. The addition of the *t*-butyl carbonium ion to the ethylene (equation (2)) is the reaction of addition of an alkyl halide to an olefin which was also discussed earlier.

The rearrangement which accompanies the exchange reaction indicated in the last equation is, as already mentioned, typical of carbonium ions containing a neopentyl system. It involves the intermediate migration of a hydride ion to form the more stable secondary pinacolyl ion which then undergoes methyl migration to yield the tertiary 2,3-dimethylbutyl carbonium ion. The latter is a tertiary carbonium ion and rapidly undergoes the hydrogen-chlorine exchange reaction to yield the *t*-butyl carbonium ion and the 2,3-dimethylbutane.



Similar rearrangements are involved in the alkylation of isobutane with propene. Again, tertiary carbonium ions can abstract hydride ions more readily from isobutane than can secondary carbonium ions and the products are 2,3- and 2,4-dimethylpentane rather than 2,2-dimethylpentane.



The carbonium ion mechanism readily explains why the primary products of the alkylation of isobutane with 1-butene in the presence of a modified aluminum chloride catalyst (aluminum chloride monomethanolate) are dimethylhexanes while those obtained with 2-butene under similar conditions are trimethylpentanes (*33*).

2,2,4-Trimethylpentane is a by-product of the alkylation of isobutane regardless of the olefin used. Its formation in the reaction with propene, for example, involves the following steps (*33*):

$$(CH_3)_3CH + C_3H_6 + H^+ \rightarrow (CH_3)_3C^+ + C_3H_8$$

$$(CH_3)_3C^+ \rightleftharpoons (CH_3)_2C{=}CH_2 + H^+$$

$$(CH_3)_3C^+ + (CH_3)_2C{=}CH_2 \rightleftharpoons (CH_3)_3CCH_2\overset{+}{C}(CH_3)_2$$

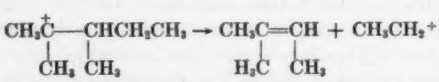
$$(CH_3)_3CCH_2\overset{+}{C}(CH_3)_2 + (CH_3)_2CH \rightleftharpoons (CH_3)_3CCH_2CH(CH_3)_2 + (CH_3)_3C^+$$

The over-all reaction is:

$$2(CH_3)_3CH + C_3H_6 \rightarrow (CH_3)_3CCH_2CH(CH_3)_2 + C_3H_8$$

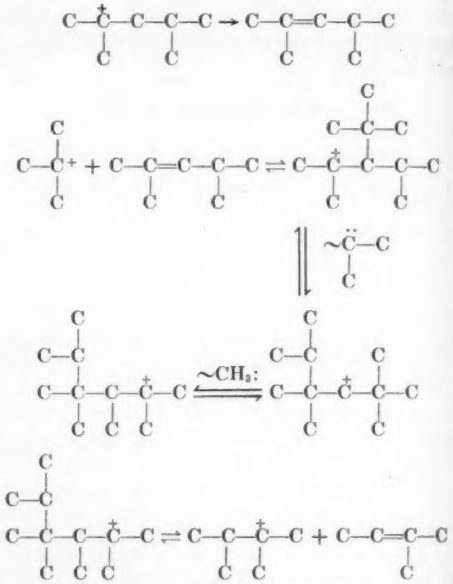
The experimental results indicate that by-product trimethylpentane and propane (or other paraffins corresponding to the olefin used) are formed in approximately equimolecular quantities.

In most isoparaffin alkylation reactions not only the so-called normal (*i. e.*, products having a molecular weight equal to the sum of molecular weights of the reacting isoparaffin and olefin) but also abnormal products (*i. e.*, products of intermediate molecular weight) are formed. Thus, isopentane and 2,3-trimethylbutane are by-products of the reaction of isobutane with propene. Their formation involves the beta-scission of intermediate carbonium ions. For example,

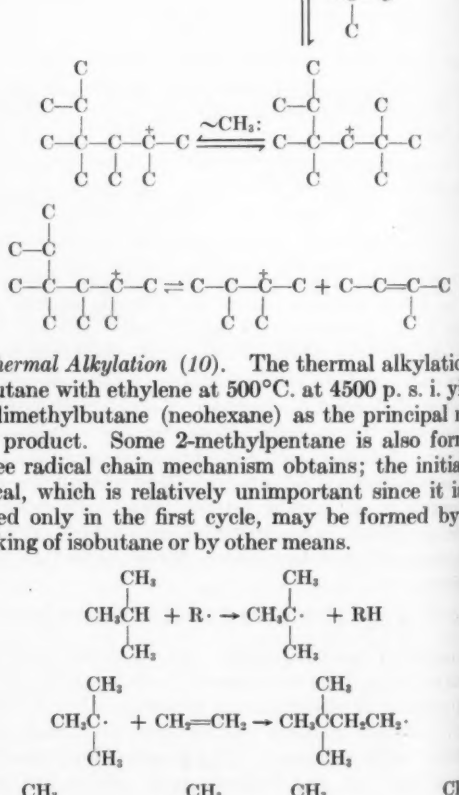


The trimethylethylene is converted to *t*-pentyl carbonium ion and thence to isopentane by the hydrogen-chlorine exchange reaction; the ethyl ion is converted either to ethane or, by loss of a proton, to ethylene which reacts with *t*-butyl ion to yield 2,3-dimethylbutane as the eventual product.

An alternative explanation (*39*) of the formation of these by-products involves the formation of an undecyl carbonium ion via the reaction of the heptyl ion and a second *t*-butyl carbonium ion:



Thermal Alkylation (*10*). The thermal alkylation of isobutane with ethylene at 500°C. at 4500 p. s. i. yields 2,2-dimethylbutane (neohexane) as the principal reaction product. Some 2-methylpentane is also formed. A free radical chain mechanism obtains; the initiating radical, which is relatively unimportant since it is involved only in the first cycle, may be formed by the cracking of isobutane or by other means.



2,2-Dimethylbutane and not 2,3-dimethylbutane is produced in the last step because, as was mentioned above, free radicals, unlike carbonium ions, do not isomerize.

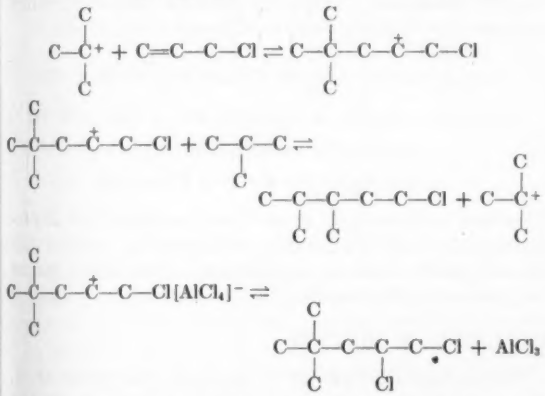
The 2-methylpentane is formed by a similar reaction

involving one of the hydrogen atoms attached to a primary carbon atom; these hydrogen atoms are less reactive but more numerous than the hydrogen atom attached to the tertiary carbon atom.

Thermal alkylation (unlike catalytic alkylation) of propane with ethylene to produce a mixture of *n*- and isopentane proceeds in favorable yield because the intermediate radicals (unlike carbonium ions) are able to abstract hydrogen from the propane readily under the alkylation conditions.

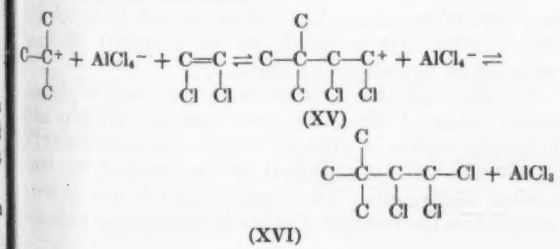
CONDENSATION OF PARAFFINS WITH CHLOROOLEFINS

Metal Halide-catalyzed Reaction. The reaction of isobutane with allyl chloride in the presence of aluminum chloride at −10°C. results in a 35–40 per cent yield of 1-chloro-3,4-dimethylpentane and a 13–15 per cent yield of 1,2-dichloro-4,4-dimethylpentane (*31*). Formation of both these products follows from the chain mechanism; it is exactly analogous to the alkylation of isobutane with propylene.



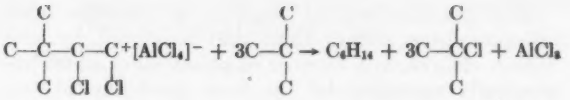
Formation of 1,2-dichloro-4,4-dimethylpentane by the reaction of *t*-butyl chloride with allyl chloride (*32*) and formation of 1-chloro-3,4-dimethylpentane by the reaction of the 1,2-dichloro-4,4-dimethylpentane with isobutane (*31*) have been shown to occur.

Polychloroolefins react similarly with isoparaffins in the presence of aluminum chloride. The reaction of *cis*- and *trans*-dichloroethylene is of particular interest since 1,1,2-trichloro-3,3-dimethylbutane is obtained in 35 per cent yield with the *cis* isomer and in less than 5 per cent yield with the *trans* compound (*34*). Difficulty of addition of *t*-butyl carbonium ions to hindered double bonds is indicated.



(XV)

(XVI)



Since no dichloro- or monochlorohexane was isolated, it may be concluded that the trichloride (XVI) and the corresponding carbonium ion (XV) are relatively unreactive toward chlorine-hydrogen exchange with isobutane because two of the chlorine atoms are attached to a primary carbon atom and the third to a secondary carbon atom in a neopentyl group. Any trichloride which does react is eventually reduced to paraffin (*i. e.*, all three chlorine atoms are successively replaced by hydrogen) by means of the hydrogen-chlorine exchange reaction. The intermediate di- and monochlorohexanes are comparatively unstable because tertiary carbon atoms are formed during the exchange reaction; for example, the trichloride is probably converted to 1,1-dichloro-2,3-dimethylbutane which isomerizes to reactive 1,2-dichloro-2,3-dimethylbutane.

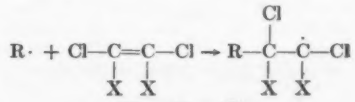
The reaction of the isobutane with chloroolefins in the presence of aluminum chloride also serves as a means of evaluating the various mechanisms which have been proposed for the alkylation of isoparaffins with olefins (*34*). None of the other mechanisms provides a satisfactory explanation for the formation of a polychloroalkane containing one more chlorine atom than the chloroolefin used.

Peroxide-induced Reaction (*37*). The condensation of saturated hydrocarbons (including propane) with polychloroethylenes may also be effected by means of peroxides. The products are chloroolefins containing one chlorine atom less than the chloroethylene used. The reaction of isobutane with *cis*- and with *trans*-dichloroethylene in the presence of di-*t*-butyl peroxide at 130–135°C. results in 35 and 38 per cent yields, respectively, of 1-chloro-3,3-dimethyl-1-butene. Condensation of propane with *sym*-dichloroethylene, with trichloroethylene, and with tetrachloroethylene proceeds in similar manner, yielding (as principal isomer in each case) 1-chloro-, 1,1-dichloro-, and 1,1,2-trichloro-3-methyl-1-butene, respectively, in 10, 31, and 27 per cent yields.

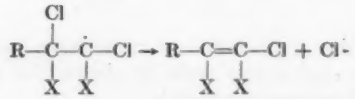
The free radical chain reaction mechanism involves chlorine atoms and alkyl radicals as the chain carriers:

$$R'OOR' \rightarrow 2R'O\cdot$$

$$R'O\cdot + RH \rightarrow R\cdot + R'OH$$



(X = Cl or H)



$$Cl\cdot + RH \rightarrow R\cdot + HCl$$

This peroxide-induced reaction of paraffins with chloroethylenes differs from that catalyzed by aluminum chloride in a number of points all of which are inherently explained by the basic principles of free radical and carbonium ion mechanisms. The peroxide-induced reaction gives good yields with both *n*- and isoparaffins; the products are unsaturated chlorides instead of saturated chlorides; and the *cis*- and *trans*-dichloroethylenes show little difference in reactivity. A significant difference between the ease of addition of *t*-butyl radicals and *t*-butyl carbonium ions to hindered double bonds is indicated by the last fact.

CRACKING OF PARAFFINS

It is well known that the catalytic cracking of paraffins yields products which are markedly different from those obtained by the thermal cracking. For example, the reaction of hexadecane in the presence of a silica-alumina cracking catalyst yields as major products compounds boiling in the C_3 to C_6 range, the compounds being largely branched-chain, little straight-chain 1-alkene boiling above the C_4 range being present (*11*). On the other hand, thermal cracking of the hexadecane yields ethylene as the single major product, with little of the aliphatic product being branched-chain, and most of the C_4 to C_{15} olefin consisting of straight-chain 1-alkenes (*11*). This difference in reaction can again be explained in terms of carbonium ions on the one hand, and of free radicals on the other.

Catalytic Cracking (*11, 41*). There is ample evidence pointing to the fact that the silica-alumina catalysts are acidic in nature. The catalysts may, for example, be titrated with potassium hydroxide and with organic bases such as quinoline. A discussion of the theories which have been advanced to explain the acidity of the catalyst is, however, beyond the scope of this paper. For present purposes, it is necessary only to assume that the reaction of the catalyst with paraffins (via incipient cracking or dehydrogenation) yields carbonium ions which start a chain reaction.

$$C_{11}H_{23}(CH_2)_4CH_3 + R^+ \rightarrow C_{11}H_{23}(CH_2)_3\overset{+}{C}HCH_3 + RH \quad (1)$$

$$C_{11}H_{23}(CH_2)_3\overset{+}{C}HCH_3 \rightarrow C_{11}H_{23}CH_2CH_2^+ + CH_2{=}CHCH_3 \quad (2)$$

$$C_{11}H_{23}CH_2CH_3^+ \rightleftharpoons \underset{(A)}{C_{10}H_{21}CH_2\overset{+}{C}HCH_3} \rightleftharpoons C_9H_{19}CH_2\overset{+}{C}HCH_2CH_3$$

$$\Updownarrow$$

$$C_9H_{19}CH_2\overset{+}{C}CH_3 \rightleftharpoons C_9H_{19}CH_2CHCH_2^+ \quad (3)$$

$$\underset{CH_3}{|} \qquad\qquad \underset{CH_3}{|}$$

$$(B)$$

$$A \rightleftharpoons C_{10}H_{21}^+ + CH_2{=}CHCH_3 \quad (4)$$

$$B \rightleftharpoons C_9H_{19}^+ + CH_2{=}\underset{CH_3}{\underset{|}{C}}CH_3 \quad (5)$$

Any of the carbonium ions present in the reaction system can start a new cycle by abstracting a hydride ion from a hexadecane molecule.

It should be noted that since primary alkyl ions readily isomerize to secondary and tertiary alkyl ions (*e. g.*, equation (3)) the electron-deficient carbon atom is unlikely to be at the end of a chain of atoms. Therefore, beta-scission of the ion must of necessity yield fragments containing at least three carbon atoms, thus explaining why the major products of catalytic cracking boil in the C_3 or higher range. Branched compounds predominate because of the ease with which the carbonium ions rearrange.

The action of aluminum chloride (promoted by hydrogen chloride) on paraffins leads to a reaction which has been termed autodestructive alkylation (*14*). It seems quite probable that this reaction involves catalytic cracking accompanied by alkylation, the alkylation proceeding via the addition of a tertiary alkyl carbonium ion to an intermediate olefin (formed by loss of a proton from a carbonium ion) in the manner described above.

Thermal Cracking (*11, 21*). The fact that ethylene is a major product of the thermal cracking of hexadecane is inherent in the principle that free radicals do not readily isomerize. This also explains why the product contains few branched-chain aliphatic compounds.

$$C_{11}H_{23}(CH_2)_4CH_3 + R\cdot \rightarrow C_{11}H_{23}(CH_2)_3\dot{C}HCH_3 + RH$$

$$C_{11}H_{23}(CH_2)_3\dot{C}HCH_3 \rightarrow C_{11}H_{23}CH_2CH_2\cdot + CH_2{=}CHCH_3$$

$$C_{11}H_{23}CH_2CH_2\cdot \rightarrow C_{11}H_{23}\cdot + CH_2{=}CH_2$$

$$C_9H_{19}CH_2CH_2\cdot \rightarrow C_9H_{19}\cdot + CH_2{=}CH_2$$

The free radicals may crack (beta-scission) or disproportionate to yield a paraffin and an olefin, or react with hexadecane to form a paraffin and a hexadecyl radical which starts a new cycle.

CRACKING OF OLEFINS

The catalytic cracking of olefins in the presence of, for example, silica-alumina catalysts, is characterized by the fact that hydrogen transfer plays a major role and is selective for tertiary olefins; furthermore, the olefins crack at a much higher rate than the corresponding paraffins (*11*). These facts follow from the mechanism of the reaction (*11, 41*). The hydrogen transfer, as has already been discussed, involves the conversion of an intermediate carbonium ion to a paraffin by abstraction of a hydride ion from another molecule of olefin or paraffin; it proceeds more rapidly with tertiary ions than with primary or secondary ions. The greater rate of cracking is due to the more ready formation of carbonium ions by addition of a proton (from the catalyst) to the olefin, whereas in the catalytic cracking of paraffins the initial carbonium ion is formed from olefins which must first be produced by incipient thermal cracking or by dehydrogenation.

On the other hand, in thermal cracking of olefins, olefins crack at about the same rate as paraffins and hydrogen transfer is a minor, nonselective, reaction (*11*). Again, this can be explained on the basis of the free-radical mechanism. The double bond is not directly involved in the formation of the initiating free radicals.

Primary and secondary alkyl radicals abstract hydrogen atoms from the allyl position in the olefins about as readily as do tertiary alkyl radicals.

ISOMERIZATION OF PARAFFINS

When pure *n*-butane is treated with aluminum chloride or aluminum bromide no reaction occurs (*25*). Even the addition of a hydrogen halide promoter will have little effect unless the reaction mixture is heated to a sufficiently high temperature to cause some cracking. On the other hand, the presence of as little as 0.01 per cent of olefin in the butane results in isomerization. The olefin presumably serves as a means of forming a carbonium ion which acts as chain initiator, and, hence, need be present in only a very minor amount. Other promoters (chain initiators) include water (*42*), hydrogen halide in substantial amount to cause cracking (about 15 mol per cent), and oxygen (*26*).

The mechanism of the isomerization of butane may be indicated as follows (*2*):

$$R^+ + CH_3CH_2CH_2CH_3 \rightleftharpoons RH + CH_3\overset{+}{C}HCH_2CH_3$$

$$CH_3\overset{+}{C}HCH_2CH_3 \rightleftharpoons CH_3\underset{CH_3}{CH}CH_2^+ \rightleftharpoons CH_3\underset{CH_3}{\overset{+}{C}}CH_3$$

$$CH_3\underset{CH_3}{\overset{+}{C}}CH_3 + CH_3CH_2CH_2CH_3 \rightleftharpoons CH_3\underset{CH_3}{CH}CH_3 + CH_3\overset{+}{C}HCH_2CH_3$$

The thermal isomerization of *n*-paraffins does not seem to have been accomplished. This is not unexpected since a free-radical mechanism would presumably be involved and, as has already been emphasized, free radicals do not readily undergo skeletal isomerization.

SUMMARY

In summary, it may be stated that reasonable explanations of a large variety of reactions have been given, based on acceptable principles of carbonium ion and free-radical behavior. It is hoped that these give a better understanding of the reactions and will help in formulating further experiments which will result in the discovery of new reactions.

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THE LITERATURE SUMMARY

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THE scientific and technical literature has today become voluminous and diffuse. Its effective utilization, however, is becoming increasingly important to industry and science, and its mastery has become a field of professional endeavor. Increasing attention is being given to devices—old and new—which aid in the translation of printed knowledge from source to prospective user. Punched cards, microfilms, microcards, and rapid scanners are becoming the order of the day. The literature researcher and the technical librarian are transforming an art into a science.

DATA TRANSMISSION

When these literature scientists have completed the *searching* part of their work, however; when, given a subject on which information is desired, they have delved into the records of the past and have come up with the existing data, they still have the task of transmitting their findings to those who have need of the information. This can sometimes be effected by simple transfer of note cards, abstract forms, or punched cards—if only one or two people must use the information obtained, and if qualifying statements and detailed indexes are not required. More often, however, the results of a literature study must be transferred in a more systematized manner, for use by a number of persons. This requires careful selection of a method of reproduction—but it first requires the preparation of the material in the most suitable form.

Several methods of data presentation are available to the literature worker, each with its particular advantages. Sometimes, a *bibliography* (indexed, or classified by subject) will suffice, where the reader, for one reason or another, wishes to follow all leads back to the primary literature. Where more information is desired, yet where equally close identification of data with source is important, use may be made of the *literature search* or annotated bibliography; items in this, also, may be classified by subject, or arranged alphabetically by author, chronologically, etc., and then subject-indexed. The abstracts or annotations included give the user more precise information about the primary literature, and aid him in selecting more accurately the references which may contain the desired data.

Finally, the literature scientist may choose or be directed to prepare a *literature summary*—literature survey, literature study, literature review, or informational survey, as it is sometimes called. Actually, the literature summary differs little in general form from the technical report or paper on original research. To prepare a literature summary properly, however, the writer: (1) must be fully cognizant of the specific needs of those who requested the information—preferably from direct conferences; (2) must have (or attain) an understanding of at least the fundamentals of his subject and a more or less extensive background in the general field of his study; (3) must be able to locate the desired information, and to recognize its importance; (4) must be able to orient his findings from the viewpoint of the prospective readers; and (5) must be able to present his facts effectively. All this requires proper scientific training, knowledge of literature science, and writing ability, plus experience in all phases of the work—which can be gained only from practice.

HANDLING OF DATA

Preparation of a literature summary requires quite a different approach from the compilation of a bibliography or search. The data located must be analyzed by the writer, not just classified for later consideration by the user. Original references must usually be scanned, in much the same manner as the ultimate reader would do it were he making the study himself.

Prior to the actual writing of the literature summary—as soon as the writer has oriented himself adequately with respect to the subject to be summarized—an outline must be prepared by which material located may be classified and cross-classified for eventual summarization. This is an important step, for without it—unless the data located are scanty—the writer will have no logical way of handling what he has found. Changes in the outline can and probably will have to be made during the course of the actual writing, but they can be handled efficiently if the data have been classified; otherwise, confusion occurs, and important facts may be lost.

Outlines are obviously susceptible to considerable variation, but in general a literature summary includes: (1) a digest or abstract of contents; (2) an introduction (which defines the scope of the summary and presents the background material); (3) a description of equipment (if the summary is on a process), physical data (if on a material), or theory; (4) a description of operations, syntheses, reactions, etc., depending on the subject of the summary; (5) a discussion of the significance of the information presented; (6) where desirable, a section containing recommendations; and (7) a bibliography.

Actual classification may be effected in any of several ways. If the writer has accumulated a file of photo-

[1] Presented at the 119th Meeting of the American Chemical Society, Boston, April 2, 1951.

copies, tear sheets, etc., of the primary literature—by no means an extravagant method unless carried to absurdity—or if he has acquired a card or note file which contains all of the data that he intends to include in his summary, he may proceed to arrange these in some logical order (perhaps in alphabetical order by authors, as they might appear in the summary's bibliography), number them, and then write the appropriate numbers under the various sections of his outline.

Alternatively, data sheets may be prepared for each section of the outline and the pertinent information then actually written thereon (with identification of source) for later regrouping and summarization. If punched cards are used to record the data collected in the course of the initial search and are coded in accordance with the outline, they need only be numbered to correspond with the bibliography, since the cards pertaining to each phase of the subject can then be sorted out as required.

Prior to and during summarization, each bit of pertinent information must be analyzed impartially and objectively. Where the data located differ (as in the case, for example, of physical data), three roads lie open: (1) reporting all the data, regardless of conflicts; (2) averaging, under certain circumstances; or (3) selectively reporting only part, on the basis of known or apparent validity. He who chooses the last-mentioned path must do so only if he is an authority on the subject or has obtained expert advice; even, then, he will do well to cite conflicting data at least by reference to source.

WRITING THE SUMMARY

When all this has been done, the literature summarizer is ready to write his review. He is now in a position similar to that of the laboratory scientist who has completed his experimentation and has made his initial correlations. Both must then present their information in the manner best suited to serve the purposes of their studies. Usually, this means the expository writing of a report or paper; in the case of the literature summary, it can even be a book.

Style and content must be carefully slanted to the audience and in accordance with the purpose involved, as must length. Differences obviously exist between the technical or scientific literature summary, the technical-economic literature summary, the "popularized" or interpretive literature summary, the patent summary, the search-summary, and the current literature summary (of any of the preceding types).

The Technical Summary. In writing the technical literature summary, the author proceeds exactly in the same manner as if he were writing any other technical paper or report. Keeping his audience in mind, he summarizes his data with attention to accuracy, clarity, brevity, consistency, and interest. When using technical terms and symbols, he defines all those which can be variously interpreted, often preparing a glossary. If he uses abbreviations, he does so consistently and in accordance with accepted practice, usually introducing uncommon abbreviations in parentheses after the full terms where they first appear.

Since the literature scientist knows that the data he is summarizing must be used by others, and be understandable to them, he is careful to introduce his subject fully and to provide background facts and theories—without, however, "writing down" to his audience. Just as in the reporting of a laboratory study, the writer, wherever possible, presents his data in tabular form, and the relations between variables and the correlation of results in the form of graphs or formulas.

As mentioned, the actual writing usually follows the path of the prepared outline.

A bibliography is an essential part of any scientific or technical summary. The literature summarizer is not the primary source for the data he presents, and the users of these data will frequently desire to review the original references for answers to additional questions, or to resolve conflicts which the summarizer did not take upon himself to decide.

The Technical-Economic Summary. The technical-economic literature summary has all of the prerequisites of the technical summary plus the slant of economic interpretation. In it, the summarizer presents his facts as background for conclusions, accurately and objectively, and with full documentation as to source.

Care should be taken to avoid "special pleading" which disregards evidence. However, the experienced summarizer can render a great service by filling in gaps from his own knowledge and by interpreting existing facts.

The "Popularized" Summary. The "popularized" or interpretive literature summary differs in scope from the technical-economic summary; it presents the summarizer's conclusions with little statement of actual data—and that only in terms which the nontechnical reader can understand. In the interpretive literature summary, the writer presents the background of the subject, the reason for the study, and the meaning of the information located. Since such summaries often find their way into technical hands for further study and recommendations, the writer must—as always—be objective and accurate, and should document his statements or at least prepare a list of selected references.

The Patent Summary. Summaries of the information contained in patents differ little from other technical literature summaries, except that the reviewer of patents must exercise unusual precautions to distinguish between legal verbosity and actual hard facts, and to differentiate clearly between closely related patents. Further, he must consider certain patents from the standpoints of validity, priority, infringement, etc. Except for their legal connotations and involved verbiage, patents may be considered as technical documents, and their data can be so summarized.

The Search-Summary. This is a hybrid form of both the literature search (annotated bibliography) and literature summary, intended for some of the purposes of both. The final report contains abstracts of the pertinent literature references, arranged in some logical

order, and numbered. Instead of preparing a detailed subject index, however, the literature worker presents the salient facts contained in these abstracts in summary form, with numerical reference to the specific abstracts that relate to each phase.

Users of a search-summary may thus quickly gain an over-all understanding of the subject in question by reading only the summary part, or they may obtain further information from the appended abstracts. Naturally, the summary must follow a logical outline or else the reader who is looking for specific information will be unable to obtain it as readily as he would from a "simple" well-indexed search.

The Current Literature Summary. Most of what has been discussed prior to this point has been concerned with summaries of broad segments of the literature—information usually obtained by searching the prior literature (at least for the last several years) on the subject in question. The summaries so prepared are as up to date as the writer can make them, and often serve to guide quick decisions, but they are not deliberately "newsy."

In recent years, increased attention has been given to the periodically prepared current literature summary, designed to keep the workers in a given field or industry abreast of the happenings which concern them, and to help executives to make decisions on an informed basis. In this category are the annual or semiannual reviews published by technical and trade journals. More to the point, however, are the monthly, bi-weekly, or even weekly summaries which at least a few groups are currently preparing.

The current literature summary differs from the current abstract bulletin in the same manner that a "summary" differs from a "search." The current abstract bulletin presents its information item by item, with detailed identification of source and rarely with any mention of conflicts even between contiguous abstracts. The current information summary, on the other hand, is prepared in semi-narrative fashion, and, even where it is strictly factual and deliberately noninterpretive, conflicts of "facts" from source to source are made to stand out clearly.

In preparing a current literature summary, the literature scientist must naturally have a broad understanding of his field or industry; otherwise, he will omit items by failing to note correlations. He must scan his selected sources—journals, newspapers, bulletins, etc.—swiftly yet thoroughly, and must organize his findings carefully—again by use of a prepared outline.

During the survey period, notes, clippings, etc., are usually classified as located and are sorted in accordance with their classifications. At predetermined intervals, a summary is then prepared, section by section, of the information obtained since the previous summary was written.

In writing his summary, the reviewer attempts to present an organized over-all picture of discoveries, statistics, statements, and events, with careful documentation as to source, either by abbreviated references following the item or sentence or by numbered references to a bibliography. Frequently, items on a given phase will not be directly related; here, the summary unavoidably resembles a sequence of abstracts. However, where several items interrelate to present information on a single aspect, true summarization must be used.

While the writer of a current literature summary generally attempts to present his facts in an absolute minimum of words, he usually also strives to summarize his findings in a readable fashion, often without the use of any but scientific abbreviations. Some writers employ a staccato style, with emphasis (by underlining, etc.) on salient points. Others, however, use continuous sentences and paragraphs, even employing, on occasion, quoted selections from the original authors' wordings, with appropriate deletions in order to accomplish the necessary brevity.

Although a given item may be publicized in several journals, its salient facts must be cited only once—which means that the writer must check items carefully to avoid such duplication. Similar but not identical papers are scanned only for incremental information, and only this is reported. In order to attain reasonable brevity, repetition of information on new theories, processes, products, or plants is held to a minimum—not an easy task since organizations are all too prone (understandably) to issue "progress reports."

Concerning new plants, for example, rumors of plans may appear in print; company announcements may be publicized; the choice of a contractor may receive attention; later reports may deal with completion schedules and trial runs; and articles may appear which describe the plant in operation. From this welter of facts, the writer of a current information summary must select the pertinent data and report them only once—in incremental fashion, if need be, and with regard to changes in plans, but with precautions against simple repetition.

Depending on the purpose of his summary, the writer may be strictly factual, deliberately refusing to comment about conflicting facts or opinions but reporting all viewpoints (with documentation to authority and to source), or he may be interpretive as well. This is a matter to be decided for each summary; however, the writer must be a real expert if he takes it upon himself to choose sides in published arguments. Moreover, he must be careful not to overemphasize the phases of a subject which happen to be of personal interest to him.

If his task is well done, the current literature summarizer renders an important service, for he adds to knowledge through the synergistic effect of bringing related information into alignment. Moreover, his objective pulling together of related facts often serves to catalyze further thinking on the part of the reader, who—lacking adequate time to read all of the necessary publications—may miss certain items and fail to connect others. Just as in the case of the preparation of other types of summaries, the work of one or two litera-

ture scientists can here be substituted effectively for the less organized, unhappy, wasteful, and incomplete efforts of many to keep abreast of their fields.

The literature summary—be it journal review, bulletin, book, or company internal report—represents the apex of achievement for the literature scientist. In its preparation he calls into play all his skills as a literature searcher; his powers of selection, differentiation, and correlation; his ability to write effectively; and his discernment as to the purpose of his study and the needs of the potential users. Information *searching* can and will be increasingly mechanized, but the end product—the information obtained—will always require informed interpretation and summation.

VLADIMIR PRELOG

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VLADIMIR PRELOG'S exceptional competence as an organic chemist is well recognized on both sides of the Atlantic. He was born at Sarajevo, Jugoslavia, on July 23, 1906. This little town in former Austria-Hungary had the distinction of being the birthplace also of World War I, for it was here that the Archduke Francis Ferdinand, heir to the Austrian throne, was assassinated on June 28, 1914.

Prelog received his early chemical education (1924–29) at the School of Chemical Engineering of the Institute of Technology in Prague. The doctoral thesis was prepared under the guidance of Emil Votoček, the well-known sugar chemist. From 1929 to 1934 Dr. Prelog worked in the laboratory of G. J. Dřiza, Prague, where fine chemicals were produced on a small scale, and he acquired skills and outlooks not usually imparted in university laboratories. In 1935 he entered academic work and served (1935–41) as lecturer and professor of organic chemistry at the University of Zagreb, Jugoslavia. Since 1942 he has been at the well-known laboratory of organic chemistry of the Eidgenossische Technische Hochschule in Zürich. Here he was promoted to a full professorship in organic chemistry in 1950.

About 170 papers record the results of his researches. These may be found in the *Collection of Czeckoslovak Chemical Communications*, Liebig's *Annalen*, *Berichte der deutschen chemischen Gesellschaft*, *Journal of the Chemical Society*, and especially *Helvetica Chimica Acta*.

His main fields of interest have been varied. They include: (*a*) heterocyclic compounds, (*b*) alkaloids, (*c*) alicyclic compounds, (*d*) biochemical materials.

In the earlier period Prelog and co-workers prepared various heterocyclic compounds and developed methods for their synthesis. In this connection derivatives of pyridine, piperidine, piperazine, and the little-known bridged heterocyclic systems, like quinuclidine, were extensively studied.

In the course of this work the compounds which are known today as the nitrogen mustards, *i. e.*, β,β-dihalogeno-dialkylamines, were prepared.

Perhaps the most outstanding results in the domain of bridged heterocycles is the resolution of Tröger's base into its optical antipodes. Not only is this the first case of the resolution of a compound with trivalent asymmetric nitrogen, but it is also the first case in which chromatography on an optically active adsorbent has been used for a preparative separation of optical antipodes.

Prelog and his research group have devoted a great deal of time to the alkaloids, and from his laboratory came several important contributions to the elucidation of the structure and stereochemistry of many alkaloids, *e. g.*, quinine, strychnine, solanidine, etc.

His work in alicyclic chemistry covers many different topics, such as: the synthesis of adamantan, which proved the constitution of this interesting and highly symmetric hydrocarbon found in Moravian naphtha; the studies on the synthesis of phenols from β-dicarbonyl compounds; and steroid and triterpene chemistry.

In continuation of the tradition of the Zürich laboratory, the many-membered ring compounds and the causes of the influence of ring size on the physical and chemical properties were investigated. These studies also contributed to the knowledge of the so-called Bredt's rule.

His studies of biochemical materials dealt with the isolation and the study of compounds present in small quantities in extracts of animal organs and in urine. A number of new substances were isolated, among them the androstane derivatives, with a musk-like odor, from boar testes, and the numerous ionone derivatives from pregnant mares' urine. The latter are the degradation products of carotinoids, and were overlooked by other investigators.

In 1949 Professor Prelog delivered the Centenary Lecture before the Chemical Society of London, and in February and March, 1950, he was Reilly Lecturer at the University of Notre Dame.

AN EXPERIMENT FOR MEASURING THE THERMAL CONDUCTIVITY OF GASES

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THE analysis of gaseous mixtures by thermal conductance depends upon the fact that the rate at which heat is lost from a hot wire varies with the composition of the gas in contact with the wire (*1*, *2*, *3*, *4*, *5*). Industrial applications of this principle have long been known, particularly for the analysis of flue gas. Although inexpensive equipment is commercially available, the method has been included in very few courses on instrumental analysis because of the difficulties encountered in preparing and handling mixtures of gases. If gas-handling equipment is used, the student must spend a large fraction of the laboratory period in becoming familiar with the methods for handling gases before getting to the portion of the experiment dealing with the measurement of thermal conductance. One way of minimizing gas handling is to take a sample of gas, such as air, and measure its thermal conductance before and after removing one component, for instance, carbon dioxide. This technique, however, suffers from the disadvantage that unknowns cannot be easily prepared. The purpose of the present paper is to show that many of these problems can be avoided by analyzing gaseous mixtures resulting from the passage of an inert "carrier" gas through a volatile liquid. Binary liquid mixtures of different compositions are easily prepared for use in making calibration curves and as unknowns.

OUTLINE OF THE EXPERIMENT

Apparatus. Figure 1 shows a simple method for setting up the apparatus, which consists of two drying

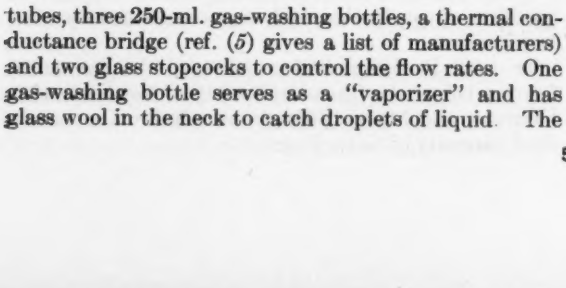

Figure 1. Gas-train for the Analysis of a Liquid by Measuring the Thermal Conductance of Its Vapor

tubes, three 250-ml. gas-washing bottles, a thermal conductance bridge (ref. (*5*) gives a list of manufacturers) and two glass stopcocks to control the flow rates. One gas-washing bottle serves as a "vaporizer" and has glass wool in the neck to catch droplets of liquid. The

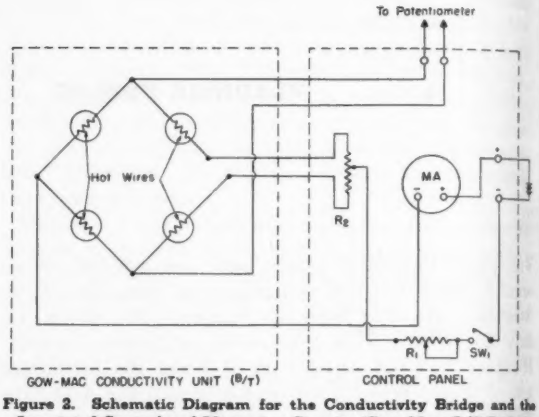

Figure 2. Schematic Diagram for the Conductivity Bridge and the Associated Control and Measuring Circuits (Gow-Mac, B/T Bridge)

R_1—current adjuster, 50-ohm radio type potentiometer
R_2—zero adjuster, 2-ohm radio type potentiometer
SW_1—on-off switch, SPST toggle
MA—d.-c. milliammeter

other two are bubble counters for gaging the rate of flow. To minimize the absorption of vapors care was exercised in making connections involving rubber tubing to insure glass-to-glass contact. The source of the carrier gas can be a compressed air line, a nitrogen tank, or room air drawn through the system by means of a vacuum line or an aspirator.

The electrical system is shown in Figure 2. The d.-c. milliammeter and the resistor, R_1, serve to control the current flowing in the circuit while the resistor, R_2, serves to balance the bridge at zero output with the carrier gas in all cells. An ordinary 6-v. lead storage battery supplies the e. m. f. The unbalance of the bridge may be read using a potentiometer or, less accurately, a microammeter (*2*).

Operation. After the electrical and gas-train connections are made and the current through the hot wires adjusted to the value recommended by the manufacturer, the bridge is allowed to warm up. Then, while passing the carrier gas alone through both sides at a flow rate of about 3 to 6 bubbles/sec. and a head of water of about 2 in. in the bubble counters, the bridge is brought to an approximate balance by adjusting R_2. The balance of the bridge is checked at approximately 5-min. intervals. After about 15 to 25 min., when the changes in balance have become less than 0.5 mv./min., one can assume, without introducing serious error in the measurements, that the bridge has reached equilibrium.

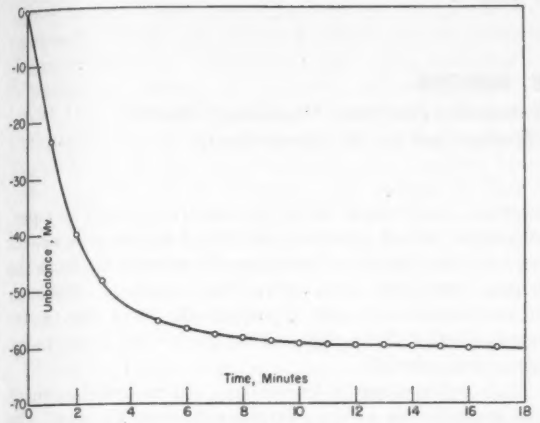

Figure 3. The Rate of Approaching a Steady Value upon Passing 400 Ml./Min. of Nitrogen through Pure Acetone (Gow-Mac, B/T Bridge)

Then the "vaporizer," a gas-washing bottle containing the liquid whose vapor is to be measured, is placed in one line and its vapor, together with the carrier gas, allowed to flow through one side of the bridge. (The flow rates through the two sides of the bridge may have to be readjusted.) Readings are then taken until a constant value is reached. A period of about 10 to 15 min. is usually required as shown in Figure 3.

Results. The extents of unbalance obtained with a number of pure solvents at about 25°C. are given in the table together with the effect of changing the carrier gas from nitrogen to dry air. Values for unbalance are given merely as an indication of the order of magnitude of the unbalance; the signs, although consistent within the set, are arbitrary. The values depend on the sensitivity of the bridge (usually defined as the millivolts of unbalance produced per 1 per cent of carbon dioxide added to air) and the ambient temperature, the latter affecting chiefly the vapor pressure of the liquid. A study over the range of 10 to 30°C. showed an increase in unbalance of about 1 mv./°C. for acetone. Ordinarily, it is more satisfactory to study the effect of a temperature change by cooling the liquid because, if it is heated more than a few degrees above room temperature, vapor will condense throughout the system.

Unbalance Produced by Vapors Obtained from Pure Liquids at About 25°C. Using a Gow-Mac Thermal Conductance Unit (Model B/T)

Compound	*Unbalance, mv.*	*Carrier gas*
Water	16.4	Dried air
	15.0	Nitrogen
Methyl alcohol	3.5	Nitrogen
Acetone	−60	Nitrogen
n-butyl alcohol	−1.61	Dried air
Benzene	−57	Dried air

Figure 4 shows a calibration curve for mixtures of acetone and water containing from 0 to 100 volume per cent of acetone. The students can prepare both the standard solutions and the calibration curve in about 90 min., following which, unknown solutions of acetone and water prepared by the instructor may be analyzed. Because of the large volume of solution used, no difficulty has been experienced from a change in the composition of a sample during an analysis.

Using the same apparatus it is possible to compare the efficiencies of different drying agents at the same or different rates of gas flow. The unbalance produced, however, is usually small and inconclusive. One can also examine the change in unbalance due to tilting (*3*) and to vibration (a sharp tap has been found to cause fluctuation as large as 0.30 mv.) of the bridge.

Possible Refinements. In the first place, because the amount of liquid vaporized is a function of its temperature and because the temperature of a volatile liquid decreases noticeably during a run, a thermostat should increase the reproducibility. Second, although most commercial bridges are designed to minimize the effect of large variations in the flow rate, one might gain precision by substituting for the bubble counters simple flow meters which are commercially available.

At the present time, work is being carried out along the lines outlined above in the hope of increasing the sensitivity and the reproducibility of the technique so that it can be applied to the analysis of a wide variety of liquids.

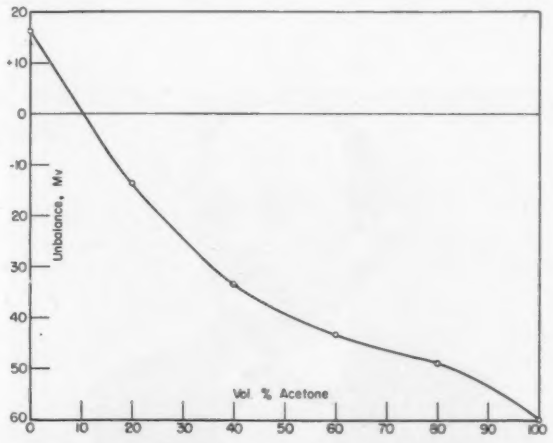

Figure 4. Calibration Curve for Mixtures of Acetone and Water Prepared by Bubbling 600 Ml./Min. of Nitrogen through Samples 2 Cm. Deep (Gow-Mac, B/T Bridge)

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(4) Palmer, P. E., and E. R. Weaver, U. S. Bureau of Standards, Tech. Paper No. 249, 18, 35 (1924).
(5) Weaver, E. R., "Thermal conductance," chap. in W. G. Berl, *Editor*, "Physical Methods of Chemical Analysis," Academic Press, Inc., New York, 1950, Vol. II.

ALEXANDER SCHMIDT[1]

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(Translated by M. Cederlund)

It is now 90 years since Alexander Schmidt announced a new theory concerning blood coagulation before the Berlin Academy of Science. At that time he was working under Hoppe-Seyler in the chemical department of the Virchow pathological institute in Berlin. This theory has withstood every subsequent criticism and even in our days it has proved useful for therapeutic work.

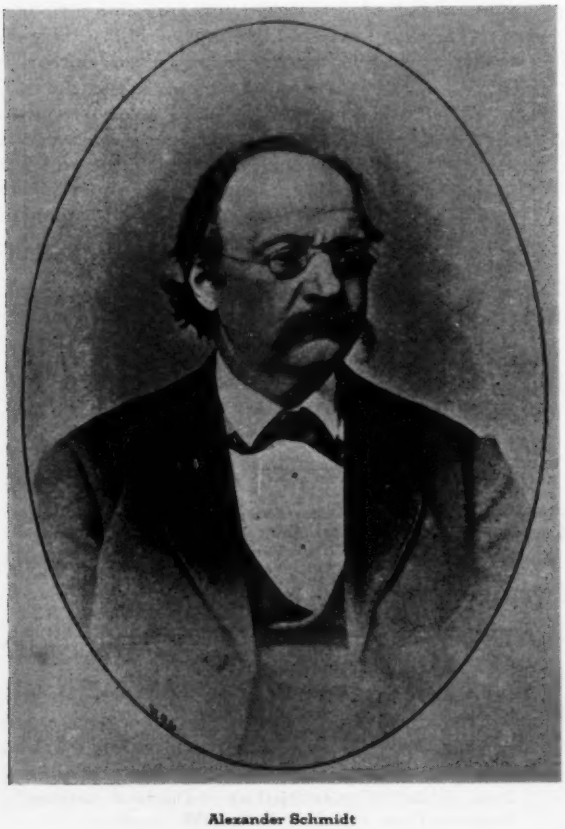
According to the theory fibrin is formed through an enzymatic denaturation of fibrinogen dissolved in the plasma of the blood stream. Thrombin, the active enzyme, is formed from an inactive precursor, prothrombin, which also is to be found in the plasma and which is activated by zymoplastic substances from the white corpuscles and from the tissues. Although thrombocytes were not known at the time, the theory is practically the same as that which we have today about coagulation.

It is interesting to know that our knowledge about the coagulation process originated with Virchow, who in 1846 explained the connection between pulmonary embolism and peripheral veinous thrombosis. He was a man of foresight and incorporated a chemical section in his new pathological institute of which the young doctor Felix Hoppe-Seyler was appointed head in 1854. During a period of 30 years Alexander Schmidt completed the study of the coagulation of the blood which he began under Hoppe-Seyler.

Schmidt belonged to an old German family. Early in the eighteenth century the Prince Elector, later King Fredrik Wilhelm I of Prussia, created the Prussian military drill system, called by Voltaire Bulgarian drill. Johan Heinrich Schmidt, a young candidate for Holy Orders, found it necessary to escape from a small village in east Prussia to the Russian side of the border in order to avoid being called to the colors. He was received by his fellow believers on the island of Ösel where he earned his bread by preaching the Gospel. (Happy days when priests fled to Russia for refuge!) His descendants followed the same vocation and one of his grandsons, Alexander, was vicar of the island of Moon with the island of Ösel as an annex. Here his son Alexander, born in 1831, grew up with his ten brothers and sisters, all of whom received their elementary education in the prosperous vicarage. Alexander received his secondary school education at Reval. In 1850 he came to the university of Dorpat to devote himself to historical studies. After having lost the sight of one of his eyes in a fencing duel of the conventional type between German students, he had to change his studies in order to make a living and took up medicine. He passed his medical examination at Dorpat.

This university has a history marked by extraordinary vicissitudes, as has the town of Dorpat itself, the cultural centre of Esthonia. Dorpat is the German name of the town, the Esthonian name is Tartu, and the Russian is Jurjef. The city, as well as the country as a whole, has had an exceedingly checkered history. It has changed hands between Russia, Sweden, Germany, Denmark, and Poland, in accordance with the changes in the power balance in the Baltic. As early as 1030 the city was taken for the first time by the Rus-

[1] The biographical data appearing in this article are taken from a memorial publication issued at the 100th anniversary of the birth of Alexander Schmidt. This was compiled from letters, notes, and oral traditions within his family and was generously presented to the author, together with the unique photograph, taken by Dr. Erhard Schmidt, professor of mathematics at the Humboldt Universität in Berlin, a son of Alexander Schmidt.



Alexander Schmidt

sians under Grand Duke Jaroslav I. It has been repeatedly plundered and ransacked and its burghers were deported to Siberia the last time, following the Russian occupation of the Baltic states during World War II. During the seventeenth century the country belonged to Sweden. As a memorial of the Swedish days, Esthonia has a Court of Civil and Criminal Appeal and a university at Dorpat. The charter of foundation of the latter was signed by King Gustavus Adolphus at his camp at Nürnberg during the 30 years war a few months before his death at Lützen in 1632.

Esthonia was ceded to Russia in 1721, but not until 1802 did Czar Alexander I revive the university at Dorpat, mainly with the aid of German scholars. During the following 90 years the university was almost exclusively German, particularly the medical faculty, although physicians were trained for service in Russia. Many famous Germans began their career as teachers there, such as the surgeon E. von Bergmann, Wilhelm Ostwald the chemist, O. Schmiedeberg the pharmacologist, Rauber the anatomist, and Ernst Kraepelin the psychiatrist. Alexander Schmidt, who hailed from Esthonia, remained at Dorpat.

After many years of study in Germany Schmidt became a lecturer at Dorpat in 1862 and became professor of physiology in 1869, a position which he held for 25 years. He published his scientific results in his pupils' doctor's treatises. In all, there were about 50 such treatises, mainly dealing with the coagulation of the blood. They were summarized in two monographs of which one, "Zur Blutlehre," was published at Leipzig in 1892, and the other, "Weitere Beiträge zur Blutlehre," posthumously at Wiesbaden in 1895.

In 1885 Schmidt was elected chancellor of the university of Dorpat for a period of four years. At about this time the Russification process had begun in Esthonia and Schmidt was the last German chancellor to be elected by the collegium.

Alexander Schmidt died in 1894 and was buried in the cemetery of St. Johns at Dorpat.

THE OVERHEAD PROJECTOR AND CHEMICAL DEMONSTRATIONS

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MANY classroom demonstrations of fundamental chemical experiments can be conducted on a scale which permits those in the back rows of the lecture hall to witness the operation. It is not probable that lecturers have resorted to wash tubs and bath tubs for these larger demonstrations, but there are times that if glass containers of comparable size were available they might have been used.

On the other hand, there are chemical phenomena which take place only on a small scale and must consequently be eliminated from demonstration experiments except for small groups. Thus, it would be almost hopeless to expect a typical member of a lecture section which exceeds a few dozen in size to observe critically a demonstration of surface tension wherein a steel needle or a razor blade is suspended by the surface film of water. In an electrolysis experiment, to report to the students in the back row that gas bubbles are forming at the anode constitutes a ridiculous situation.

In order to solve this problem the writer has adapted an overhead projector[1] to several chemical demonstrations, some of which are described below. The advantages of this type of presentation are at once obvious. In classes numbering over 200 students, all members can easily observe the demonstration as it is projected on the screen even though the room is partially illuminated. This permits the student the opportunity of taking notes at the same time that the demonstration is being projected on the screen. There is a minimum amount of disruption to the course of the lecture because the projector is on the lecture table and the lecturer who operates the projector remains in visible contact with the class at all times.

Noller[2] has suggested the use of the overhead projector for the demonstration of optical activity. There

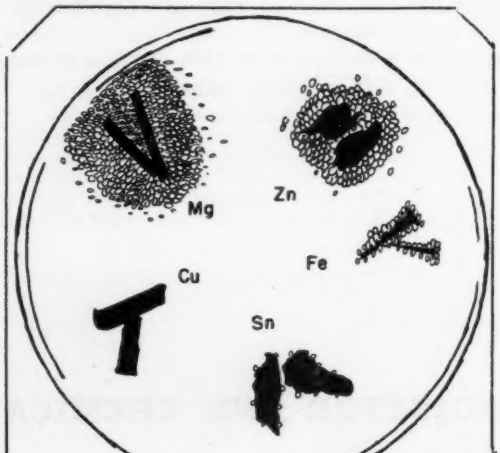
[1] Overhead projectors are available from the Charles Beseler Co., American Optical Co., and Bausch & Lomb Optical Co.

[2] NOLLER, C. R., J. CHEM. EDUC., 26, 269 (1949).

are undoubtedly others who have used this medium for specific demonstrations, but there appears to be a great number of commonly used demonstrations which can be performed in this manner.

DEMONSTRATION EXPERIMENTS

(1) *The Activity Series for Metals.* Symbols for common metals such as magnesium, zinc, iron, tin, and copper are written with wax pencil on the bottom of a crystallizing or culture dish. Dilute hydrochloric acid is then placed in the dish to a depth of 5 mm. Into the dish on the projection table are dropped small pieces of the appropriate metals, whereupon hydrogen bubbles form at a rate relative to the activity of the metal. The figure shows the projected image.



The Relative Activity of Certain Metals in Displacing Hydrogen from Dilute Hydrochloric Acid

(2) *Electrolysis of Water.* A miniature Hoffman apparatus with electrode chambers about 10 cm. long can be easily constructed using glass tubing of oval cross section. Circular cross-section tubes filled with liquids will appear as solid shadows on the screen. The arms and reservoir are mounted at 45° to normal so that the image on the screen will show these parts. In performing this experiment the gas bubbles which form at the electrodes will appear on the screen as large as 20 cm. in diameter. In the traditional manner, organic dye indicators may be used which will indicate the acidic and basic nature of the electrolyte at the cathode and anode, respectively.

(3) *Random Motion of Gas Molecules (Kinetic-Molecular Theory).* A dozen or more pieces of metallic sodium approximately 2 mm. in diameter are dropped into a crystallizing dish which contains a few ml. of water. The degree of activity of this reaction causes the metal to melt into spheres almost immediately, and to travel at speeds of 10 to 20 cm. per sec. across the surface of the water. At the edges of the dish, the water meniscus provides a suitable barrier which turns the globules of sodium back an angle of reflection apparently equal to the angle of incidence. The turbulent wake caused by this motion leaves a record of the path which the metal sphere follows, but this wake soon subsides so that the greater portion of the surface of the water remains undisturbed. The presence of phenolphthalein in the water produces a very colorful wake which adds considerably to the spectacular nature of this experiment.

(4) *The Action of Metal Couples.* Pieces of zinc and copper screen are placed in dilute hydrochloric acid in a crystallizing dish. Hydrogen gas is evolved at the zinc screen until the two metals are placed in contact with each other. Then the gas bubbles form on the copper. Metal in the form of screen has been found advantageous because of the greater degree of visibility provided by this form in contrast to solid pieces of the metals.

(5) *The Relative Strength of Acids.* Five small crystallizing dishes containing dilute solutions of HCl, H_2SO_4, CH_3COOH, citric acid, and boric acid are placed on the projector. When small pieces of zinc are added to each dish the relative activity of each acid is easily deduced on the basis of the quantity of hydrogen bubbles which appear on each metal.

(6) *The Qualitative Aspects of Optical Activity.* A crystallizing dish containing a solution of an optically active compound is sandwiched between two sheets of Polaroid. Orientation of the upper sheet will readily show the effect of rotation of the plane of polarized light by the optically active compound.

(7) *Electrochemical Experiment.* Not only small scale cells may be projected but also the electrical measuring instruments, if constructed on transparent plastic bases; all information concerning the experiment becomes directly available to each member of the class. Wiring diagrams of the electrical circuits can be made an integral part of the actual apparatus.

Many other lecture experiments can be performed in this manner. The color response of indicators to acids and bases, the formation of insoluble products, the speed of chemical reactions as a function of state of subdivision, and the exhibition of molecular models are examples. In setting up experiments with the overhead projector it is well to bear in mind that the apparatus for the most part must be transparent and that the view on the screen will be as if one were to look directly downward at the experiment.

It has recently been suggested by Kharasch[3] that lantern slides can be used to an advantage in organic chemistry courses. The overhead projectors now available not only project the standard slide from the lecture table, but permit the use of slides which are made on cellophane, Kodapak, or other transparent media. Diagrams, charts, graphs, etc., up to 7 × 7 inches can be easily reproduced by simply tracing the original onto the transparent sheet by means of a wax pencil. The storage of slides of this type resolves into the use of an ordinary letter file.

[3] KHARASCH, NORMAN, J. CHEM. EDUC., **28**, 280 (1951).

GERMAIN HENRI HESS AND THE FOUNDATIONS OF THERMOCHEMISTRY[1]

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THERMOCHEMISTRY had its beginning in the studies of Lavoisier and Laplace which established the fact that the heat absorbed in a reaction is equal to the heat liberated in the reverse reaction. This work was not followed up, and chemists in the first half of the nineteenth century were mostly concerned with the analysis and reactions of inorganic and organic compounds. Heat was a subject which was left to the physicist. The work of Germain Henri Hess was therefore the more original, since he pioneered in a field which his contemporaries were neglecting almost completely. His own training had done nothing to prepare him for thermochemical studies, since it was exactly that of all the chemists of his day. In fact, he was well known among his colleagues for a wide variety of studies in both inorganic and organic chemistry before he undertook his work in thermochemistry. Today, however, the situation is reversed, and his other work is forgotten. Only his name is now remembered, and this only in connection with his chief generalization—the Hess law.

Hess was born in Geneva on July 26, 1802 (*1*), but at the age of three he was taken to St. Petersburg, where his father was a teacher, and almost all of his life was spent in Russia. In 1822 he entered the medical faculty of the University of Dorpat, then one of the leading universities of Russia. Although his studies were chiefly medical, he found time to obtain a thorough training also in chemistry and geology. His thesis for the degree of Doctor of Medicine was on a chemical topic, "Studies of the chemical composition and medicinal action of the mineral waters of Russia."

He obtained his degree in 1825, and then decided to go to Stockholm to study chemistry in more detail with Berzelius. On December 9, 1825, the latter wrote to Wöhler, "Osann, who is coming here from Dorpat, has sent me another student, Dr. Hess, a young man of much promise. He has a good head, seems to have a foundation of knowledge, much attentiveness, and a particular zeal" (*2*). Hess's stay in Stockholm was brief, however, for on January 13, 1826, Berzelius wrote, "Hess, who came here from Dorpat, has gone away again, compelled to return by domestic affairs. I was well pleased with him" (*3*). In spite of the shortness of his stay with Berzelius, Hess remained friendly with the great Swedish chemist throughout his life, and was often mentioned in the letters Berzelius wrote to his various correspondents.

Courtesy of the Edgar Fahs Smith Collection, University of Pennsylvania

Germain Henri Hess

The interests of Berzelius in mineral chemistry and chemical analysis were also those of Hess, and all his early work was done in these fields. In this he was following in the path of most of the Russian chemists of his time, for, after the days of Lomonosov, theoretical studies had been practically abandoned in Russia, and almost all attention was centered on the discovery of new minerals.

Following his return to Russia, Hess accompanied a geological expedition to the Urals, and then settled in Irkutsk, where he took up a medical practice. In addition to the duties of his practice, which included studies on the prevention of eye disorders which were very common in the region, he found time to continue his geological and chemical studies. As a result of his descriptions and analyses of minerals and waters from various parts of Russia, he was elected an adjunct member of the Academy of Sciences of St. Petersburg in 1828, and in 1830 he was chosen as an extraordinary Academician. Upon receiving this honor, he abandoned his medical practice and settled in St. Petersburg, where he devoted the rest of his life to chemistry.

[1] Presented before the Division of the History of Chemistry at the 119th Meeting of the American Chemical Society, Boston, April 3, 1951.

The following ten years were filled with activity. He signalized his membership in the Academy with a summary of his work at Irkutsk, "On the common salt of the Irkutsk Government," in which he showed that the sodium chloride from this area contained a high proportion of potassium, magnesium, and aluminum. He also undertook a study of the composition of the water of the Neva River which flows through St. Petersburg. He discovered a number of new minerals, and proved the composition of the oxide of cobalt Co_3O_4, a study which was later confirmed by Berzelius. His work on the natural resources of Russia led him to analyze natural gas from Baku, and various resins and waxes. He thus became interested in methods of organic analysis and in problems of organic chemistry. His main contribution in this field was his study of the oxidation of sugars by nitric acid, which led to his discovery of saccharic acid.

When Hess came to St. Petersburg, the Technological Institute had just been organized, and he was appointed to a Commission to plan the course in practical and theoretical chemistry. He was appointed to the chair of chemistry in this school soon afterward. As new institutions of higher learning were organized, he began to teach in these also. In 1832 he joined the faculties of the Mining Institute and the Chief Pedagogical Institute, and in 1838 he added the course at the Artillery School to his other teaching duties. In the Chief Pedagogical Institute, which trained teachers for most of Russia, one of his students was A. A. Voskressenskiĭ, who later became Professor of Chemistry in the same Institute, and in this capacity taught chemistry to Mendeleev.

As a result of his teaching duties, Hess began to work on a textbook of chemistry soon after he reached St. Petersburg. This work, "Fundamentals of Pure Chemistry," was published in two volumes by the Academy of Sciences from 1831 to 1833. It did much to establish a rational nomenclature for chemistry in the Russian language. In 1834, Hess published a one-volume abridgement to serve as a more usable textbook, and this went through seven editions, the last of which appeared in 1849. It became the standard Russian text in chemistry until the appearance of Mendeleev's two texts, "Organic Chemistry" in 1861, and "Fundamentals of Chemistry" in 1868–70.

In addition to his research and teaching duties, Hess also found time to serve on government commissions relating to the teaching of chemistry and the establishment of a water supply for St. Petersburg. In 1834 he was made an ordinary Academician of the Academy of Sciences.

In 1838, Hess began the series of studies on which his fame chiefly rests. In a paper, "The evolution of heat in multiple proportions" (*4*), he showed that the various hydrates of sulfuric acid could be detected by the heat evolved in their formation. In 1840 he published a full statement of his law, both in French in the *Bulletin of the Academy of Sciences of St. Petersburg*, and in German in *Poggendorf's Annalen der Chemie und Physik* (*5*). He showed that not only for the formation of hydrates of sulfuric acid, but also for the heat of formation of ammonium sulfate in water solution and the neutralization of hydrochloric acid by sodium and potassium hydroxides, the amount of heat developed was always the same, regardless of whether the process went directly or proceeded through a number of intermediate steps. This "law of the constant summation of heat" obviously showed how to determine the heat of reactions which had not been measured directly, and it lies at the basis of all later work in thermochemistry. It is a remarkable fact that, although it clearly depends on the law of conservation of energy, this law had not been announced at the time Hess was making his studies, and was not actually stated until two years later, by Meyer in 1842.

Hess fully appreciated the significance of his law, and did not hesitate to make practical applications of it. An excellent illustration is found in a letter he wrote to Arago in 1840 (*6*). At that time, the heating power of coal was estimated by determining the amount of oxygen required to burn it. The English chemist, Ure, observed that the more hydrogen a coal contained, the less heat it gave off, and so the oxygen method did not give an accurate measure of the heating power. Ure explained this by assuming that the vapors formed in burning the coal absorbed heat. Hess pointed out the error of this assumption, and wrote:

> The sum of the heat corresponding to a certain amount of water and carbonic acid which we suppose arises from combustion, *being constant*, it is evident that if hydrogen is found previously combined with carbon, this combination cannot have occurred without evolution of heat; this amount, already eliminated, cannot be recovered in the quantity evolved by the defined combustion. There results in practice the simple rule: that *a combustible compound always evolves less heat than do its elements taken separately*. . . . I have the full conviction that we will have a precise idea of chemical phenomena only when we succeed in indicating in our formulas the ratios of heat relations as we indicate today the relative numbers of ponderable atoms; at least thermochemistry promises to disclose the still secret laws of affinity.

Continuing his studies, Hess was led in 1842 to his second major law, the "law of thermoneutrality." This states that in exchange reactions of neutral salts in water solution, no heat effect is observed. Hess himself was unable to explain this fact in a satisfactory manner, and it was not until the announcement of the Arrhenius theory of electrolytic dissociation in 1887 that the explanation became apparent.

After this time, Hess became less active in research, and progressively fewer papers appeared from his pen. He served actively on committees of the Academy of Sciences, and helped other chemists to obtain recognition for their work. It was largely through his interest that Karl Klaus, a rather obscure chemist at the University of Kazan, was able to gain recognition for the discovery of ruthenium. Hess and Fritsche, another Academician, were instrumental in securing for Klaus the Demidov prize, a major scientific award in Russia at that period.

Hess continued his teaching activity, and besides his

work in the various institutions, he was entrusted with the training of the Czarevich Alexander in chemistry. In 1838, Berzelius wrote Wöhler a description of a visit to his laboratory by the Czarevich, who was then touring Sweden (7). Alexander told Berzelius that Hess had taught him chemistry from the Berzelius "Lehrbuch," and Alexander was "fairly well versed in it." In parting, the royal visitor gave Berzelius a valuable ring as a gift. Berzelius closed his letter with the remark, "I would gladly have such a visit every week."

By 1848 the health of Hess began to give way, and he resigned most of his teaching duties. He made an inspection of the newly opened sugar factories in the south of Russia in that year, but this was almost his last public duty. He died on November 30, 1850, at the early age of 48.

His work was not continued by any other Russian chemist, and, in fact, thermochemistry was generally neglected for another decade. Then began the elaborate series of investigations of heats of reaction by Berthelot in France and Thomsen in Denmark. The mass of data accumulated by these investigators confirmed the truth of the Hess law again and again, but almost nothing was said of Hess himself, and chemists forgot the man whose work lay at the foundation of all the new material which was appearing.

The work of Hess was once more brought to light by Wilhelm Ostwald, who had begun his own career at Riga and Dorpat, and had probably there heard of Hess. In his "Textbook of General Chemistry," published in 1887, Ostwald began the section on thermochemistry with a full account of the contributions of Hess in this field, and later he reprinted the most important papers of Hess in his series *Klassiker der exacten Wissenschaften* (8). It is strange, however, that, unlike most of the volumes in this series, the volume on Hess does not contain any biographical material. Thus, while the contributions of Hess are now recognized, and his name is firmly attached to the law which he first announced, the details of his life have been difficult to obtain.

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(2) WALLACH, O., "Briefwechsel zwischen J. Berzelius und F. Wöhler," Leipzig, **1901**, Vol. 1, p. 98.

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(5) *Ibid.*, **50**, 385 (1840).

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A COMPACT HYDROGEN SULFIDE GENERATOR

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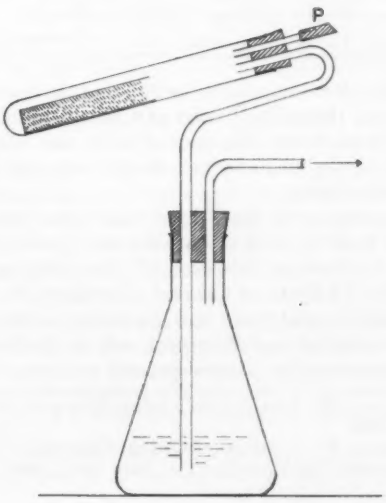
IN RECENT years many laboratories have been using a powdery mixture called "Aitch-Tu-Ess" which, when heated, evolves hydrogen sulfide in sufficient volume for the needs of a student. The usual arrangement of the apparatus for the purpose includes an iron laboratory stand with clamp, to hold a test tube in which the Aitch-Tu-Ess is placed. In addition, a wash bottle should be added, through which the evolved gas passes to the test solution.

A student in one of the writer's classes (Mr. V. Kocenko) devised for his own use the arrangement shown, which eliminates the stand and clamp. The operation of the generator is essentially the same as for the equipment usually employed. The test tube is heated, either holding the burner in the hand or placing it, with properly adjusted flame, on the table beside the flask. When heating is stopped the rubber policeman, *P*, is removed to prevent sucking water back into the tube during cooling. A widemouthed bottle may be used instead of the flask.



A COMMON MISUNDERSTANDING OF HESS'S LAW

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In a series of measurements having wide general significance, G. H. Hess in 1840 determined the amount of heat evolved when sulfuric acid is neutralized by solutions of alkalies. Since the addition of sulfuric acid to water releases heat even though no alkali is present, it proved to be of great interest to know how the heat of neutralization varied when the acid was first diluted with different amounts of water. The measurements were all made at essentially atmospheric pressure. The data led to Hess's "law of constant heat summation."

The original statement is "Wenn eine Verbindung stattfindet, so ist die entwickelte Warmemenge constant, es mag die Verbindung direct oder indirect und zu wiederholten Malen geschehen." A familiar translation is "The amount of heat evolved during the formation of a given compound is constant, independent of whether the compound is formed directly or indirectly in one or in a series of steps."

This statement preceded a recognition of the law of conservation of energy and may be regarded as one of the bases for that law. The work of J. R. Mayer and J. P. Joule on the conservation of energy came later.

The following numbers taken from Hess's papers are typical.

Typical Data from Hess's Study of the Neutralization of Sulfuric Acid by Ammonia

Substances added together			*Heat evolved on adding*		*Total heat*
Acid	*Water*	*Base*	*Water*	*Ammonia*	
H_2SO_4	...	$2NH_3$	...	595.8	595.8
H_2SO_4	H_2O	$2NH_3$	77.8	518.9	596.7
H_2SO_4	$2H_2O$	$2NH_3$	116.7	480.5	597.2
H_2SO_4	$5H_2O$	$2NH_3$	155.6	446.2	601.8
				Average	597.9

The fourth column in the table gives the heat evolved on diluting the sulfuric acid and the fifth column the heat evolved when the diluted acid was neutralized. The sum of the heat effects is nearly constant as shown in the last column.

Hess's papers on the subject have been reprinted as Number 9 of Ostwald's *Klassiker der Exakten Wissenschaften.*[1] A clear discussion of the work appears in Ostwald's "Outlines of General Chemistry."[2]

In recent years there has grown up a tendency to quote Hess's law out of context and to attribute to it, rather inaccurately, a more general meaning than was originally intended. Applied to determinations carried out in the manner used by the original author, the law is both correct and useful. According to the law, one can, for example, combine data on the heat of formation of carbon dioxide and on the heat of combustion of carbon monoxide to calculate the heat of formation of carbon monoxide from the elements. Direct measurement of this heat cannot be made with satisfactory accuracy. Such applications of the law are both correct and valuable.

New types of thermal measurement have been developed since Hess's time and they require a more careful statement and a more careful use of Hess's law than seems apparent in several popular American textbooks. In particular, bomb calorimetry and the investigation of reversible reactions have extended the whole basis of thermochemistry. In one current book, we find the statement: "In any series of chemical or physical changes the total heat effect is independent of the path by which the system goes from its initial to its final state." Were the word "energy" substituted for "heat," the statement would be correct. As given it is wrong. In another book, we read: "The heat change in a chemical reaction is the same whether it takes place in one or in several stages. This means that the net heat of reaction depends only on the initial and final states and not on the intermediate stages through which the system passes." The use of the word "heat" makes the statement false. The net heat of reaction depends not only on the initial and final states of a system but also on the intermediate stages.

Serious contradictions arise at once if one applies the versions of Hess's law quoted above. These are typical of what one can read in a great many present-day books. There are exceptions, of course. The statement in Taylor and Glasstone's "Treatise on Physical Chemistry" is correct[3] and so is the clear statement in Macdougall's "Thermodynamics and Chemistry."[4] Suppose one tries to apply the familiar form of Hess's law to the synthesis of water.

$$2H_2(g.) + O_2(g.) = 2H_2O(g.)$$

When carried out in an open vessel, the heat of reaction at 100°C. is 116,210 cal.[5] for every two mols of water formed, the starting materials and the product all being gases at 1 atm. pressure. The "law" tells us that

[1] Ostwald, W., *Klassiker der exakten Wissenschaften*, No. 9, Leipzig, 1890.

[2] Ostwald, W., "Outlines of General Chemistry," translated by J. Walker, The Macmillan Co., New York, 1890, p. 209.

[3] Taylor, H. S., and S. Glasstone, "Treatise on Physical Chemistry," 3rd ed., D. Van Nostrand Co., Inc., New York, 1942, Vol. 1.

[4] Macdougall, F. H., "Thermodynamics and Chemistry," John Wiley & Sons, Inc., New York, 1939.

[5] Taylor, H. S., and H. A. Taylor, "Elementary Physical Chemistry," D. Van Nostrand Co., Inc., New York, 1942, p. 72.

if we start with two mols of hydrogen and one of oxygen, all at 100° and 1 atm. and end with two mols of steam at 100° and 1 atm., a very definite amount of heat will be released. It is not so.

We may prefer to effect the combination in any of many other ways. Two particular paths, in addition to the constant pressure combination, may be represented diagrammatically as follows:

Path A: 2 mols of H_2, 100°, 1 atm.; 1 mol of O_2, 100°, 1 atm. $\xrightarrow{\text{reaction in bomb calorimeter}}$ 2 mols of steam, 100°, 2/3 atm. $\xrightarrow{\text{compression}}$ 2 mols of steam, 100°, 1 atm.

Path B: 2 mols of H_2, 100°, 1 atm.; 1 mol of O_2, 100°, 1 atm. $\xrightarrow{\text{compression}}$ {2 mols of H_2, 100°, 3/2 atm.; 1 mol of O_2, 100°, 3/2 atm.} $\xrightarrow{\text{reaction in bomb calorimeter}}$ 2 mols of steam, 100°, 1 atm.

In neither of these cases will the amount of heat evolved add up to 116,210 cal. For the sake of simplicity, we assume that hydrogen and oxygen and steam are all ideal gases at 100°. The assumption of ideal gas behavior means that the same amount of heat will be liberated in the bomb calorimeter reaction along path A as along path B, the internal energy of the gases being independent of the pressure. Let us designate this heat as Qv. The value of Qv may be derived in the following way. For the constant pressure reaction,

$$q = \Delta E + P\Delta V,$$

where q is the heat evolved, ΔE is the decrease in "internal energy" ("chemical energy"), P is the pressure, and ΔV the volume decrease accompanying the reaction. In the bomb reaction no mechanical energy is received or contributed by the hydrogen-oxygen system, so that the heat of reaction will be ΔE.

$$\Delta E = Qv = 116{,}210 - p\Delta V$$
$$= 116{,}210 - 740 = 115{,}470 \text{ cal.}$$

In addition to Qv, the heat of compressing 2 mols of an ideal gas from 2/3 atm. to 1 atm. will be liberated along path A and the heat of compressing 3 mols of gas from 1 atm. to 3/2 atm. will be liberated along path B. The total heat evolved along the two paths then will be (ideally)

Path A: $Qv + 2\,RT \ln \frac{1}{2/3} = 115{,}470 + 600 = 116{,}070$ cal.

Path B: $Qv + 3\,RT \ln \frac{3/2}{1} = 115{,}470 + 900 = 116{,}370$ cal.

We may contrast these figures with the heat liberated in the constant pressure process, namely, 116,210 cal. Depending entirely on the path, therefore, one might observe any one of a great many values for the heat of formation of steam at 100°, the steam having the same final pressure as the hydrogen and oxygen initially.

An even more impressive difference in the heat of formation of steam would be observed if the hydrogen and oxygen were allowed to combine at the electrodes of a reversible electrochemical cell. Some of the energy of the combination would appear as electrical energy and the heat of synthesis of the water would be considerably less than in the irreversible combination. Actually the heat of formation in the reversible process at 1 atm. pressure would be about 8600 cal., a figure quite different from the others given above.

It is apparent that the common form of Hess's law applies to the type of experiment carried out by Hess. It is wrong to state the law without such qualification. While the language is not that of the original, one might preserve the accuracy of Hess's law and indeed its spirit by stating that the internal energy change accompanying a chemical reaction is independent of the path and depends only on the initial and final states.

It is usual to introduce Hess's law in the freshman chemistry course and there arises the problem of presenting the law in a form that is both simple and correct. We can hardly do better than quote Taylor's statement of Hess's law as a solution to the problem (ref. 3). "The heat evolved in any chemical reaction at either constant pressure or constant volume is independent of the manner in which the reaction is achieved, whether in one or many steps."[6]

To the student who may inquire as to the reason for the restriction "at constant pressure or constant volume," it may be pointed out that in chemical reactions not only is "chemical energy" converted into heat[7] but "mechanical energy" may be converted as well. Two terms, namely, ΔE and W together determine q, not one alone. Only if the amount of mechanical work is made definite can the sum of the internal energy change plus the mechanical energy change, which together determine the heat evolution, be specified. At constant volume or at constant pressure the total mechanical work converted to heat is definite, so the total heat effect will have a fixed magnitude. At constant volume, regardless of the steps, the total conversion of mechanical work into heat is zero while at constant pressure, regardless of the steps, the total conversion of mechanical work into heat is $P\Delta V$. These statements apply provided there are no frictional or other constraints on the system which would lead to the degradation of mechanical, electrical, and similar forms of energy to heat. In the presence of such constraints degradation to heat may occur with an accompanying alteration in the total heat effect.

If Hess's law is to be discussed at all, it should be given in correct form. In the process of presenting the law it would seem appropriate to mention the fact that changes in mechanical and electrical energy may occur incidentally in chemical reactions. These changes superimposed on the changes in internal or chemical energy determine the heat effects. The latter changes are themselves, as a rule, not sufficient to determine the magnitude of the observed heat evolution.

[6] Actually this statement stands in need of the further restriction that no other constraint is imposed on the system, as explained below.

[7] Possibly into other kinds of energy depending on the experimental circumstances surrounding the reaction.

ENERGY REQUIREMENTS FOR NUCLEAR TRANSFORMATIONS[1]

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THERE are several conservation requirements that must be met in nuclear reactions. The familiar Einstein relation $E = Mc^2$ is rightfully considered the Rosetta stone for all nuclear transformations. Nevertheless the conservation of charge, angular momentum, *i. e.*, spin, and linear momentum[2] are additional factors that should be considered. Most textbooks in chemistry neglect the principle of conservation of linear momentum. In overlooking this principle, textbooks frequently give false estimates of energy requirements for simple reactions. Friedlander and Kennedy's text[3] is one of the few in which the principle of conservation of momentum is properly considered.

Historically this principle was first applied to atomic and nuclear investigations by Rutherford[4] to derive his famous formula for the scattering of alpha particles. Since that time momentum considerations have figured in many nuclear reactions, particularly those studied in a Wilson Cloud Chamber.[5] Bethe[6] cites the case in which the mass of ${}_2He^5$ is determined by measuring the kinetic energy and momentum of the nuclei in the reaction

$$ {}_3Li^7 + {}_1H^2 \rightarrow {}_2He^4 + {}_2He^5 $$

To illustrate the application of momentum conservation we may consider the simple case of a lithium target which is bombarded by alpha particles to give the reaction

$$ {}_2He^4 + {}_3Li^7 \rightarrow ({}_5B^{11}) \rightarrow {}_5B^{10} + {}_0n^1 + Q \qquad (1) $$

The symbol Q stands for the sum of the kinetic energy of the products minus the sum of the kinetic energy of the reactants and represents the mass equivalent of the energy released (or absorbed) in the transformation. Using the values for the atomic masses for the substances in equation (1) a value of $Q = -2.87$ m. e. v. may be obtained.

The usual impression students get from this information is that if one were to bombard the lithium with alpha particles having a kinetic energy of 2.87 m. e. v. the transformation in equation (1) would be possible. Consideration of the principle of the conservation of linear momentum would prohibit this reaction. The kinetic energy of the alpha particle must be appreciably greater to permit equation (1) to proceed from left to right. The energy in excess of 2.87 m. e. v. may be calculated by writing equation (1) as

$$ {}_2He^4_m \xrightarrow{P_m} {}_3Li^7_M \xrightarrow{P_{M+m}} ({}_5B^{11})_{M+m} \xrightarrow{P} \begin{matrix} \nearrow {}_0n^1 \\ \searrow {}_5B^{10} \end{matrix} \qquad (2) $$

Here P_m is the linear momentum of the alpha particle; the lithium nucleus is considered *initially* at rest. The symbols m and M represent the mass of the alpha and lithium nuclei, respectively. The momentum $P_m = mv$ where v is the velocity of the alpha particle. Both ${}_0n^1$ and ${}_5B^{10}$ receive a push in the direction of P when they fly apart. The same is true of the compound nucleus $({}_5B^{11})$.

Let the kinetic energy of m and $M + m$ be T_m and T_{M+m} respectively. Then[7]

$$ Q = T_m - T_{M+m} $$

or

$$ Q = \frac{P^2_m}{2m} - \frac{P^2_{M+m}}{2(M+m)} \qquad (3) $$

P_{M+m} is the momentum of the compound nucleus. However, because the lithium is initially at rest we have, as a result of the conservation of momentum,

$$ P_m = P_{M+m} = P $$

Thus

$$ T_m = Q + \frac{P^2}{2(M+m)} \qquad (4) $$

or

$$ T_m = Q + \frac{P^2\mu}{2m} $$

where

$$ \mu = \frac{m}{M+m} $$

Hence

$$ T_m = Q + T_m\mu $$

or

$$ T_m = \frac{Q}{1-\mu} \qquad (5) $$

[1] Presented at the 117th Meeting of the American Chemical Society, Philadelphia, April, 1950.

[2] For a discussion of the validity of Newton's Third Law as applied to atomic and nuclear phenomena see GERJUOY, E., *Am. J. Phys.*, **17**, 477 (1949).

[3] FRIEDLANDER, G., AND J. W. KENNEDY, "Introduction to Radiochemistry," John Wiley & Sons, Inc., New York, 1949.

[4] RUTHERFORD, E., J. CHADWICK, AND C. D. ELLIS, "Radiations from Radioactive Substances," Cambridge University Press, 1930.

[5] For example, see POLLARD, E. C., *Proc. Roy. Soc.*, **141**, 375 (1933).

[6] BETHE, H. A., "Elementary Nuclear Theory," John Wiley & Sons, Inc., New York, 1947.

[7] The absolute value of Q is used here.

For the nuclear reaction in question $m = 4$ and $M = 7$ so that $T_m = 11/7Q$. Since $\mu < 1$ equation (5) may be written as

$$T_m = Q(1 + \mu + \mu^2 + \ldots) \quad (6)$$

For the special case where $m \ll M$ it will be seen that

$$T_m \cong Q \quad (7)$$

Equation (7) appears to be the relation that is usually given regardless of the relative values of m and M.

It will be seen then that in equation (1) the energy of the incident helium nucleus must be at least 11/7 times the value of Q if the reaction is to occur, barring other factors such as potential barrier effects, etc.

Hence the calculated energy requirements for a nuclear reaction may be incorrect if the principle of the conservation of momentum is neglected.

SOME LECTURE DEMONSTRATIONS IN GENERAL CHEMISTRY

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THE following demonstrations have been found of value in presenting to beginning students concepts of the atomistic nature of matter, the controlled experiment, and the solubility product.

(1) The concept of the atomistic nature of matter can be demonstrated by (*a*) peeling an orange, separating the segments, seeds, etc., and observing the internal structure of particles within particles, (*b*) comparing the pouring of sand (with its visible particles rolling over one another) with the pouring of water (with invisible particles), and (*c*) comparing the decrease in total volume resulting on admixture of equal volumes of absolute alcohol and colored water with a similar change in volume resulting from pouring sand into a container holding oranges.

(2) The value of a control or blank experiment and the importance of introducing singly, as variations from the blank, only those factors under investigation can be illustrated by means of the Landolt (or iodine clock) reaction. A blank Landolt experiment is carried out at room temperature according to customary laboratory procedure for the reaction. Two subsequent runs are made at about 35°C. with (*a*) minor variations in apparatus (*e.g.*, method of stirring, size of beaker) plus 10 g. of anhydrous sodium sulfate and 3 ml. of oil of wintergreen added per liter of total solution, and (*b*) no variations from the blank other than temperature. Experiment (*b*) illustrates the regular positive temperature coefficient of rate of reaction.[1] The former experiment, however, exhibits a lower velocity than for the blank due to an overwhelming retardation of the reaction by the sodium sulfate, which not only decreases the rate of the Landolt reaction at constant temperature[2] but also produces a negative temperature coefficient.[3] The oil of wintergreen and changes in apparatus serve only as extra variables and appear to have no effect on the rate of reaction. By performing the multivariable experiment immediately after the blank, one can provoke some student discussion on the validity of the result as a measure of the effect of increased temperature on the rate of reaction.

(3) That the concentration of bromide ion enters the solubility product expression for lead bromide to a higher power than the concentration of lead ion does can also be effectively illustrated by means of a lecture demonstration. A saturated solution of lead bromide is prepared by mixing 250 ml. of 1 *M* lead nitrate with 500 ml. of 1 *M* sodium bromide and removing the precipitate by suction filtration. Treatment of one-half of the filtrate with 100 ml. more of the lead nitrate solution produces almost no additional precipitate, whereas addition of 100 ml. more of the sodium bromide solution to the other half of the filtrate gives extensive additional precipitation within a matter of seconds. This result proves rather surprising to students who are encountering chemical equilibria for the first time, inasmuch as they almost invariably predict that the greater amount of precipitate should form from the added lead nitrate solution due to the smaller volume of this solution required in the original stoichiometric mixture.

[1] Convenient reaction times (about 20, 38, and 17 seconds for the three runs, respectively) are obtainable from mixing equal volumes of the following two stock solutions: (*a*) 25 ml. of 5 *N* sulfuric acid, 25 ml. of 1 per cent (by weight) soluble starch solution, and 400 mg. of c.p. potassium iodate per liter of solution; and (*b*) 620 mg. of c.p. anhydrous sodium sulfite per liter of solution.

[2] Unpublished results from the Chemical Laboratories of Indiana University.

[3] SERABAL, A., *Z. Elektrochem.*, 21, 461–3 (1915).

WHAT THE CHEMISTRY TEACHER SHOULD KNOW ABOUT PATENTS

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IN PROPORTION to the nearly universal need, the future chemists and chemical engineers are taught less of patent law probably than of any other subject. Those who are to succeed in their chosen profession and actually push back the frontiers of knowledge may reasonably expect to make inventions. They have a right to some education in advance with respect to patents, from teachers having a speaking acquaintance at least with the subject.

For this reason, the teacher should know some patent law and, first of all, what types of discoveries are patentable.

PATENT HISTORY

As early as the third century B.C. the importance of incentives for even simple inventions was recognized. In Sybaris, one of the towns of Southern Italy, a confectioner or cook who "invented any peculiar and excellent dish" received the right to make the dish exclusively for a year, as an inducement to others "to labor in excelling in such pursuits" (*1*).

From such a rational beginning, the patent system degenerated into abuse in barbarian lands. For centuries, British monarchs, for example, issued patents on almost any right they wished to bestow. Henry II, after 1100 A.D., granted to a picturesque Irish outlaw a patent on the right to raise a volunteer army in Britain to fight in Ireland (*2*). Queen Elizabeth issued to crown favorites patents on such old products as wheat starch and playing cards (*3*).

Such abuses disappeared largely with enactment of the Statute of Monopolies in 1623. This in effect limited the granting of patents to *inventions* or *new manufacture within the realm.*

The United States and also most foreign countries now require both newness and invention.

The United States Constitution (*4*) gives to Congress the power "to promote the progress of science and the useful arts by securing for limited times to authors and inventors the exclusive rights to their respective writings and discoveries." The period of time set by Congress for inventors is 17 years from the date of issuance of the patent. The purpose is encouragement of invention.

REQUIREMENTS FOR PATENTABILITY

A principal feature of our present patent laws is the limitation of subject matter to be patented to the following classes:

Patentable classes	*Examples*
Composition of matter	Water-soluble vitamin A composition
Method	Making nylon or scrambling radio signals
Machine	Crystallizing machine or corn harvester
Article of manufacture	Asbestos shingle or cigarette lighter
Ornamental design	Pattern of table silverware
Plant produced asexually	Sport variety of apple tree

All other classes of subjects are unpatentable. Such excluded subjects are systems of doing business, seeds, plants produced sexually, as by cross pollination, treatment of the human body, new use of an old composition without any modification except as a method, production of fissionable material and use of it or of atomic energy in a military weapon, abstract principles of nature, and mathematical formulas.

The additional requirements are as follows:

(1) *Utility* for a purpose neither immoral nor injurious to the health or good of society. Utility ordinarily is easily proved. A new chemical compound will be understood to have utility as an intermediate in the manufacture of another compound at least. A toy has utility. So does a cosmetic, in the sight of the law. The green fluorescence of a lubricating oil of a large manufacturer, on the other hand, was questioned. Was it useful? The fluorescence was visible to the buyer as identification but served no function during use of the oil. However, utility was finally held to be present. A method of manufacturing a useful article or composition has utility.

(2) *Novelty* or newness over all that was previously known.

(3) *Technical requirements as to dates.* To be patentable, to an applicant, the invention must *not* have been:

(*a*) known or used by another before the date of invention by the applicant.

(*b*) published in the United States or abroad or in public use in the United States for more than twelve months before the date of filing of the United States application for patent.

(*c*) patented in a foreign country on an application filed more than twelve months (six months in cases of designs) in advance of the United States filing date.

(4) *Invention.* Mixing sugar with a material to sweeten it is not invention; the result is obvious. Protecting a material against oxidation by admixing sugar, on the other hand, is invention if such protection was previously unknown and particularly if the result is contrary to what was to have been expected. Prescriptions, recipes, and those other compositions in which

each ingredient serves only its usual function are not inventions. To be invention a discovery requires the exercise of ingenuity above the reasonable skill of the worker in that art.

TESTS FOR INVENTION

Invention defies accurate definition. After thousands of decisions on invention, it remains still as mysterious as that ever-elusive luminous body rising through the fog above the moor, the tantalizing will-o'-the-wisp that many claim to have seen but no one has either apprehended or comprehended.

Invention is in reality *a discovery that is unobvious to the expert in the art.*

Perhaps the greatest of all inventions is the scientific method. Within the walls of the research laboratory, the individual rises to heights of creativeness heretofore unapproached (*5*). As a result, the requirement "unobvious to the expert in the art" is continually rising, hand in hand, with the increasing skill available for making inventions.

As tests for invention, the inventor should ask himself the following:

Does the discovery produce a result unobvious to the mythical "expert in the art" knowing all that has gone before but possessed of only usual reasonable experimental skill? For invention, the answer must be "Yes."

In case of doubt as to the above, would this expert have been expected to try what the inventor has done and, having tried it, to have found the same result? The answer must be "No."

Has there been long delay in meeting a public need after the necessary materials became available? Has there been commercial success where others have failed? Affirmative answers to these two questions are indications but not proof of invention. They cannot swing the balance to the side of invention when invention, for other reasons, is considered clearly absent (*6*).

Improvements. Nearly all patentable discoveries are improvements on something previously known. Examples are the argon filled light bulb over the previous vacuum bulb of Edison, one variety of rayon over another, plasticized lacquer films over unplasticized, ethoxyethyl esters over the corresponding ethyl esters, and television over radio, although TV uses many of the radio techniques and patents. To be patentable, the improvement must show an unexpected advantage.

New Use. Consider the case of a recently discovered insecticide, AB, of epoch-making importance. The compound in crystal form was old. To claim "*An insecticide* consisting of the compound AB" is not permissible as the law has been recently interpreted (*7*). For patentability of the new use, it was necessary to add after "compound AB" the following: "in the form of a powder and a diluent therefor" (*8*). The compound had not been previously powdered or mixed with a diluent and the inventor's contribution was so outstanding that only slight modification of the old composition was required to make the use as insecticide patentable.

A new use frequently is patentable as a *method.* Thus the use of the compound AB in any form might have been claimed as follows: "The method of killing insects which comprises contacting them with the compound AB."

Substitution of Materials. As to substitution of materials, the incandescent light cases provide a series of decisions to the effect that substitution of materials may constitute invention. Each of the following changes in turn were held to be invention: platinum for carbon filaments in the light bulb, pressed tungsten wire for platinum, and wrought tungsten for pressed tungsten. The changes gave an added function such as longer life, lower cost per candle power, and increased brilliancy.

Likewise, in make and break contacts for automobile ignition systems, change of material to tungsten was held patentable; tungsten contact points, in oxidizing, give a coating that shakes off during use of the automobile so as to expose a fresh surface continuously.

Would it be invention to substitute steel by stainless steel and find only decreased corrosion, low melting paraffin by a higher melting paraffin and find only that the resulting composition has a higher softening point, or cotton fibers by asbestos and find only decreased flammability? The answer, of course, is "No."

Omitting or Adding an Ingredient. Subtracting or adding to a composition or other class of subject is frequently invention.

Assume that *removing* carbohydrate from a hormone is found to be necessary before the hormone is dissolved in a solvent and emulsifier. A patent cited by the Patent Office against the application for patent on such a method teaches, let us say, the necessity of *adding* carbohydrate before solution in the same solvent with a somewhat different emulsifier. The discovery here of the benefit from removing the carbohydrate is contrary to the teaching of the art. It is invention.

Adding an ingredient also is invention when the result produced is not obvious. Adding oat extract to ice cream is not invention if the addition gives only an oat flavor. But the oat extract also prevents development of rancidity in the fat present. Such a beneficial property was not to have been expected and its discovery when made was invention.

Proportions. Proportions of materials used provide a basis for patentability when the proportions are critical. This is true particularly when the difference from previously used proportions is large and a different function is served by the new proportions.

In concentrating minerals by flotation, change in the proportion of frothing oil from the 4 per cent of oil previously used to "a fraction of one per cent" came before the Supreme Court. The use of the smaller proportion of oil was more economical. Also it caused the minerals to float instead of sink as in the earlier process. The Court held the patent valid (*9*).

Untried Inventions. These are patentable. One who speculates only, however, may miss the most important point of novelty that experiment would reveal.

PATENT POLICIES

When a large investment is to be based on a discovery of doubtful patentability, it is good policy to file the application while continuing experiments to find some unexpected result, even in a secondary feature. If the product is a fireproof blanket, for example, and the fireproofness was to have been expected, mildew resistance, although of relatively minor importance, may be the unexpected result giving patentability. On the other hand, if a product has excellent patentability but poor prospects for use, it should not be patented unless funds are plentiful.

Publication for a year before filing an application is a bar to patenting in the United States. Publication for a single day is a bar in many foreign lands. Policy as to filing both domestic and foreign applications should be determined, therefore, before publication.

The proposed publication is best submitted to the patent attorney before it is released. He will note especially whether the chemist has made the development seem so logical as to destroy the lack of obviousness necessary for patentability. To have stumbled onto something by accident or found an unexpected result may be the last act that the chemist would like to admit. Yet this is an excellent route to patentable invention.

Consider the case of Charles Goodyear. The proper manner of describing the circumstances of his discovery of rubber vulcanization would have been about as follows: "I had been trying unsuccessfully for many weeks to obtain a hard rubber by exposing to sunlight a mixture of rubber with sulfur (*10*). Only slight hardening occurred. By accident I dropped a sheet of the rubber and sulfur mix on a hot stove. To my surprise, the sheet was cured."

CONCLUSION

Invention did not stop with Charles Goodyear. It is proceeding at an increasing rate. Knowledge of the law of inventions is more vital for technical workers now than ever before.

The leaders in education as well as industry should impart to young men that patent consciousness which is so conspicuously lacking in most new graduates. These leaders, knowing the fundamentals here stated, should also be able to create a desire to learn more of this interesting and important subject.

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A NEW METHOD FOR FINGERPRINTING DIAMONDS

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An optical instrument, invented by Bruce Eytingĕ and developed by the Manhattan Research Laboratories of New York, can, in a matter of seconds, photographically record the light that is reflected from the back or pavilion facets of a diamond. A spot pattern results, characteristic for each stone tested.

The instrument (see Figure 1) consists of a metal cylinder at the base of which is a fixed light source in the form of a six-volt lamp. Parallel light is projected by a simple lens system to the diamond which is centered and placed, table facet down, on an optical glass flat. A short distance below the glass plate is a holder on which is placed a sheet of photographic film. Ordinary commercial film of the kind used for back-reflection X-ray work can be employed, since this type already has a circular hole in the center.

Each pavilion facet reflects the projected light at a fixed angle, which in turn is impinged on the film. On developing the film, a number of spots, more or less irregularly distributed, result. Since the facets of no two

diamonds of the same weight and cut are ever fashioned absolutely (mathematically) alike, no two fingerprints of identical pattern are possible. If the stone is a different one, or if the original gem has for some reason been recut or relapped, a wholly different reflection spot pattern will be obtained.

Should the same stone be photographed at some later date, it is only necessary to superimpose one negative over the other to obtain a "mesh" or coincidence of spots. A ground-glass viewer of the kind used to examine X-ray radiographs is a valuable aid in examining fingerprint negatives.

An exposure of four seconds is all that is required to obtain a spot pattern. The instrument will handle faceted stones other than diamond. Its most important use, however, is in the positive identification of diamonds.

Diamonds of different cut, such as the brilliant, emerald, and the marquise, give very distinctive fingerprints, as illustrated by Figure 2. These spot patterns are useful in disclosing whether or not a diamond is properly proportioned.

Double refraction in a gemstone can be photographically recorded with rather spectacular results. Figure 3 illustrates the marked double refraction of Zircon. To obtain this type of photo a different technique must be employed. In this case, the stone is placed table facet up on a small metal cylinder which can be adjusted

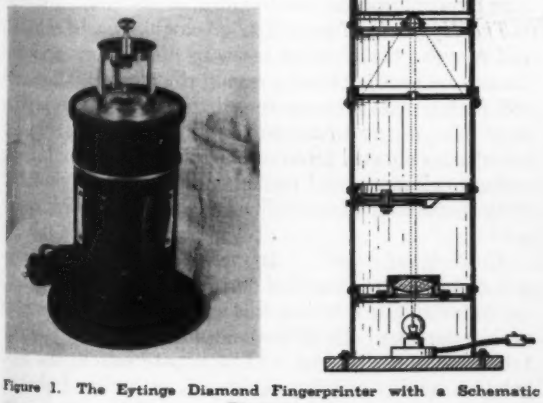

Figure 1. The Eytinge Diamond Fingerprinter with a Schematic Diagram

to support the gem at the girdle edge. (An old photographic diaphragm is very well suited for use as a stone holder.) The cylinder must be carefully centered on the optical glass flat, over the opening through which the light beam is projected; otherwise distortion of the double refractive effect may occur. To achieve the maximum double refraction, such as is produced by Zircon, it is necessary that the culet facet of the gem be parallel to the table facet. The film in this instance is placed above the table facet at a distance of one or

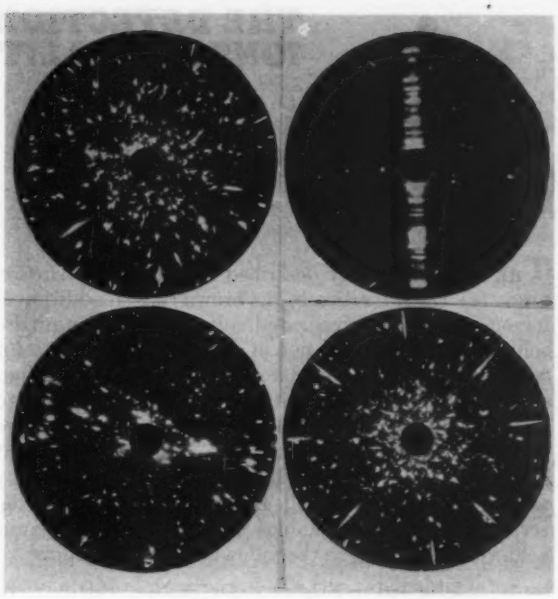

Figure 2. Diamond Fingerprints

Upper left: brilliant cut. Lower left: marquise cut. Upper right: emerald cut. Lower right: properly proportioned brilliant.

several inches. A few seconds' exposure suffices to register the amount of double refraction. A doubly refractive stone should be examined optically, or its indices of refraction carefully measured on a refractometer using sodium light, to insure the obtaining of the gemstone's maximum double refractivity. The table facet of some doubly refractive gemstones may prove to be cut normal to an optic axis, in which case no double refraction in this direction is possible.

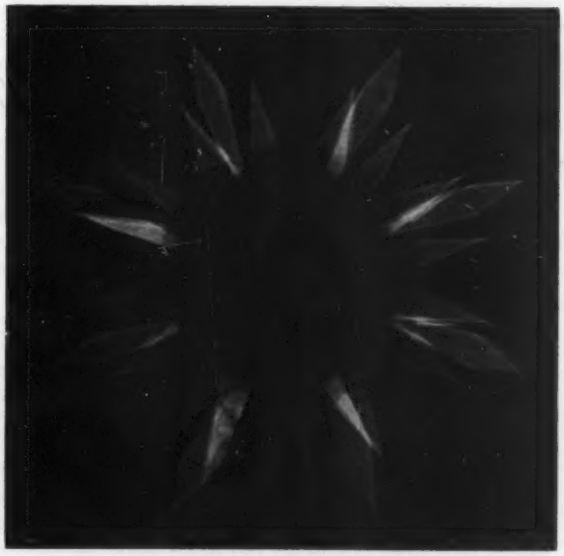

Figure 3. Double Refraction Recorded from a Brilliant-cut Zircon

THE DETECTION OF NITRITE AND SULFAMATE IONS IN QUALITATIVE ANALYSIS

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THE most commonly described tests for the nitrite ion are: (*a*) the formation of the brown ring on addition of ferrous ion and concentrated sulfuric acid to a nitrite solution; (*b*) the formation of a starch-iodine blue color when iodide ion is added to acidified nitrite solution; and (*c*) the evolution of nitrogen when urea is added to an acidified nitrite solution. The test (*a*) is neither specific, sensitive, nor very well adapted to student semimicro use. Test (*b*) is very nonspecific since any weak oxidizing agent will give the color reaction described. Test (*c*) resembles in principle the sulfamic acid-nitrite reaction as the following equations show.

$$NH_2SO_3H + NO_3^- \rightarrow HSO_4^- + N_2 + H_2O \quad (1)$$

$$2H^+ + NH_2CONH_2 + 2NO_2^- \rightarrow 3H_2O + 2N_2 + CO_2 \quad (2)$$

The reaction represented by (2) is less sensitive to the presence of nitrite and preacidification of the solution is necessary, meaning that an unwanted anion must be added to the solution. A secondary test is suggested by the formation of the acid sulfate ion in equation (1). On adding barium chloride reagent to the reaction mixture white barium sulfate is formed if nitrite is present. The insolubility of barium sulfate in hydrochloric acid differentiates this precipitate from barium carbonate which would form if the carbonate anion were present.

EXPERIMENTAL

Sensitivity of the Sulfamic Acid Test for Nitrites. A positive but very weak test (effervescence) for the nitrite ion is obtained when three drops of a 1 *M* solution of sulfamic acid (or several small crystals) are added to 1 ml. of test nitrite solution containing 0.01 mg. of $NaNO_2$ (10 μg.). The test both with regard to effervescence and formation of barium sulfate is unmistakable at a concentration of 0.07 mg./ml. (70 μg.). At comparable concentrations of sodium carbonate and of sodium sulfite (about 0.001 *M*) no effervescence is noted. The interference of carbonate and sulfite ions is further discussed below.

Tests for Interfering Ions. A number of anions were studied in order to determine which would interfere with the nitrite test using sulfamic acid. The following have been noted as not constituting any interference when present in reasonable concentrations: $C_2H_3O_2^-$, F^-, Cl^-, Br^-, I^-, AsO_4^{---}, PO_4^{---}, SO_4^{--}, $C_4H_4O_6^{--}$, $C_2O_4^{--}$, SCN^-, CN^-, SiF_6^{--}, $Fe(CN)_6^{---}$, $Fe(CN)_6^{-4}$, I_2(aq.), and Br_2, (aq.).

Anions which in an acid solution are better oxidizing agents than nitrous acid are not listed above since a reaction would occur in conjunction with the sulfamic acid-nitrite reaction. A number of oxidizing agents were tested for their reaction with sulfamic acid. No reaction was noted at ordinary temperatures for the following: CrO_4^{--}, MnO_4^-, BrO_3^-, IO_3^-, IO_4^-, and BiO_3^-.

Anions which do react with sulfamic acid, giving an effervescence, or which might conceivably be considered as interfering with the nitrite ion, are: OCl^-, OH^-, S^{--}, SO_3^{--}, $S_2O_3^{--}$, CO_3^{--}. The last mentioned interferes the most seriously.

The Hypochlorite Ion, OCl^-. A relatively slow reaction occurs between hypochlorous acid at high concentrations and sulfamic acid. The rate of reaction is much more rapid at elevated temperatures. In practice the reaction between hypochlorous acid and the nitrite ion to form nitrate ion would take place more rapidly than the reaction between sulfamic acid and hypochlorous acid. The relative rates of the three reactions mentioned may be represented as: $(NO_2^- + HSO_3NH_2) > (NO_2^- + HClO) > (NH_2SO_3H + HClO)$. The rapid and vigorous reaction between nitrites and sulfamic acid cannot be mistaken in the cold for the slow hypochlorous acid sulfamic acid reaction.

The Hydroxide Ion, OH^-. A highly basic solution will prevent the reaction between nitrite ions and sulfamic acid for the obvious reason that the sulfamic acid will preferentially be converted to a salt. The proton must be present to initiate reaction (1). A solution basic to bromcresol green or phenolphthalein containing sodium sulfamate and sodium nitrite gives a slow but definite reaction when acidified to about 1 *M* with acetic acid.

The Sulfide Ion, S^{--}. In view of the fact that hydrogen sulfide may be evolved on the acidification of a concentrated sulfide solution, this ion must be considered as an interference. Its detection and removal by precipitation are no problem. The nitrite ion in an acid solution readily oxidizes sulfide ion to sulfur; however, not before the nitrite ion would react with sulfamic acid if the latter were added as a test reagent.

The Sulfite, SO_3^{--}, and Thiosulfate, $S_2O_3^{--}$, Ions. Both of these ions will decompose in an acid solution. The effervescence is slight and would not be mistaken for that due to the nitrite ion. More serious is the reaction between the sulfite ion (or sulfur dioxide) and the nitrite ion in an acid solution (see equation (3)).

$$2\,HNO_2 + SO_2 \rightarrow SO_4^{--} + 2H^+ + 2NO \quad (3)$$

Whether or not the sulfite ion will completely mask the nitrite test will depend on the rapidity with which the

sulfamic acid test is made and the relative amounts of sulfite and nitrite ions. Unless the nitrite ion is present in nothing more than a trace, it is doubtful that either the sulfite or thiosulfate ions would constitute a serious interference.

The Carbonate Ion, CO_3^{--}. The carbonate ion interferes seriously with the nitrite test. If a positive limewater test is noted on the gases evolved when sulfamic acid is added to an unknown solution, a carbonate ion concentration of at least 10 mg./ml. is indicated. Since the nitrite ion is detectable down to a concentration of 0.07 mg./ml., it is possible to dilute a few drops of unknown solution to a point where effervescence is still noticeable without giving a positive limewater test. If sulfate-free sulfamic acid crystals (or solution) are used in the test, a few drops of 0.1 *M* $BaCl_2$ are added to the unknown solution to act as a supplementary test for nitrite. The appearance of white barium sulfate, insoluble in HCl, in conjunction with a positive limewater test on the evolved gas, indicates the presence of both the nitrite and carbonate ions. One operation is needed then, to test for both ions. Since an unknown solution might well contain the sulfate ion, it would be desirable to make the nitrite test on the filtrate after the removal of anions forming insoluble salts with a heavy metal and the barium ion.

Test for the Sulfamate Ion by the Nitrite Ion. In most anion analysis schemes used at the present time, the sulfamate ion would be included in a soluble group since all normal salts of sulfamic acid are soluble. The only ions which would interfere with the sulfamate ion (or sulfamic acid) test using nitrite ion as a test reagent would be those ions which react very rapidly with nitrites to give an effervescence. The proton decomposes nitrites rapidly. Since the solution must be at a pH of 2–3 for the nitrite-sulfamic acid reaction to take place, the original unknown is acidified with 4 *M* acetic acid. Any effervescence occurring on this acidification is allowed to go to completion. Carbonates and sulfites are destroyed under these conditions. Two drops of 0.1 *M* $NaNO_2$ reagent solution added to the acetic acid solution give an effervescence if the sulfamate ion is present. The rate at which nitrites are decomposed by an acetic acid solution is too slow to constitute an interference by nitrite reagent. The minimum concentration of sulfamic acid detectable is about 1.6 mg./ml.

Detection of the Nitrite Ion Using Sulfamic Acid. Three drops of the original unknown solution are diluted to about 3 ml. (approximately 50 to 1). Three drops of this diluted solution are treated with several small crystals or a drop of 1 *M* sulfamic acid solution. An evolution of an odorless gas strongly indicates the presence of the nitrite ion. Add two drops of 0.1 *M* barium chloride reagent solution to the solution after effervescence has stopped. The appearance of a white precipitate of barium sulfate, insoluble in dilute hydrochloric acid, further indicates the presence of the nitrite ion. The reactions are represented as:

$$NO_2^- + NH_2SO_3H \rightarrow N_2 + HSO_4^- + H_2O$$

$$HSO_4^- + Ba^{++} \rightarrow BaSO_4 + H^+$$

The supplementary barium sulfate test on the original unknown solution is obviated if the original unknown contains the sulfate ion. The dilution of the original solution (50 to 1) permits the detection of nitrite at a concentration of 10 mg./ml. in the presence of 100 mg./ml. of carbonate. If the original solution is saturated or nearly saturated with carbonate (*e. g.*, sodium carbonate) effervescence will be noted which will mask the nitrite test. The carbonate ion is readily detected by adding sulfamic acid reagent solution or crystals to the undiluted unknown and testing the evolved gas for carbon dioxide. If a positive test is obtained for carbonate, barium ion is added to precipitate barium carbonate and the nitrite is tested for on the filtrate as described above without the dilution step.

Detection of the Sulfamate Ion by the Nitrite Ion. Three drops of unknown solution are treated with barium ion and a heavy metal ion (*e. g.*, cadmium) to remove possible interfering ions such as carbonate, sulfide, and sulfite. A drop of phenolphthalein or bromcresol green indicator is added to the filtrate, now free of interfering ions, followed by the addition of two drops of 0.01 *M* sodium nitrite test solution. Dilute (4 *M*) acetic acid is added in a three-drop excess beyond the assumption of the acid color of the indicator. A rapid effervescence of nitrogen indicates the presence of the sulfamate ion. Since excess barium ion may be assumed as being present from the previously mentioned removal of insoluble barium salts, a white precipitate of barium sulfate should form if the sulfamate ion is present.

An alternate procedure is to add 4 *M* acetic acid dropwise to three drops of the original unknown solution prior to the addition of the sodium nitrite solution. Any effervescence is allowed to continue to completion. The interfering carbonate, sulfite, and sulfide ions are eliminated. The sodium nitrite test solution is then added. Effervescence at this point is attributed to the reaction between sulfamic acid and nitrite ion.

THOMAS GRAHAM'S STUDY OF THE DIFFUSION OF GASES

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GRAHAM published his first paper on gases in 1826 (*10*, *19*) and in 1829 the first on the diffusion of gases appeared (*15*). Since 1826 he had been teaching chemistry in a very meagerly equipped private laboratory in Glasgow. In 1829 he became the lecturer on chemistry in the Mechanics Institute. In 1830 he was named professor of chemistry in Anderson's College. Though there was no endowment attached to this position and the laboratory was furnished with very little apparatus, it was here that he carried on the experiments on the diffusion of gases that established "Graham's law" and it was here also that he worked on the constitution of the phosphoric acids and their salts that led to the concept of polybasic acids (*14*).

Thomas Graham, son of a Glasgow merchant and manufacturer, was born December 20, 1805. He was sent to an English preparatory school in 1811; in 1814 he transferred to the high school, entering the University of Glasgow in 1819, before reaching the age of 14. Here the excellent teaching of Thomas Thomson (*17*), professor of chemistry, and Meikleham, professor of natural philosophy, aroused his interest in scientific work, especially molecular physics. Despite the desire of his father that he enter the ministry, Graham decided to devote his life to the pursuit of science. After receiving an M.A. degree in 1824 he went to Edinburgh where he spent the next two years under Hope (*16*), the discoverer of strontium. He then returned to Glasgow where he taught chemistry in his own laboratory. In 1829 he was appointed lecturer on chemistry at the Mechanics Institute and the next year he attained a professorship of chemistry at Anderson's College. In 1837 he became professor of chemistry at University College in London and he held this position until his appointment as master of the mint in 1855. Here he remained until his death at his home in Gordon Square, London, on September 11, 1869.

Prior to the publication of Graham's first paper on the diffusion of gases, the experimental information known amounted to little more than the well-established fact that gases of different kinds, when brought into contact, do not arrange themselves according to their density, the heaviest undermost and the lightest uppermost, but they spontaneously diffuse, mutually and equally, through each other, and so remain in an intimate state of mixture for any length of time (*1*). The earliest observations on this subject were those of Priestley who had noticed that when gas was transmitted through stoneware tubes surrounded by burning fuel, the gas escaped outward into the fire, while at the same time the vapors evolving from the fire penetrated into the tube, although the gas within the tube was in a compressed state. Dalton next discovered that any two gases, when brought into direct contact, tended to flow through each other, even in opposition to their weight. Dalton thus concluded that particles of different gases offered very little resistance to each other. Döbereiner (*18*) had accidently observed that as hydrogen escaped from a cracked glass jar the water from the pneumatic trough rose into the jar. He incorrectly ascribed this phenomenon, however, to capillary action, and supposed that hydrogen only was attracted by the fissures, and escaped through them because of the extreme smallness of its atoms (*9*).

As can be seen from the above information, Graham had very little worth-while background material upon which to lay the foundation of his work. The apparatus with which he began his experiments was of the utmost simplicity.

A cylindrical vessel, shaped like a test tube, about 9 inches in length and 0.9 inch internal diameter, was divided into 150 equal parts, and provided with an accurately ground glass stopper, through which was fitted, also by grinding, a short piece of stout tube, 0.12 inch in bore and 2 inches in length, and bent in the middle at right angles.

Although the stoppers fitted precisely, the joinings were sealed as an additional precaution.

Graham's first experiment was the diffusion of different gases into atmospheric air. The vessel described above was filled in succession with various pure gases and supported in a horizontal position upon a frame within a box, with the end of the bent tube pointing upward when the contained gas was heavier than air, and downward when the gas was lighter, to avoid any tendency of the gas to flow out of the receiver. After the gas had been allowed to diffuse into the air for a certain length of time the receiver was transferred to the pneumatic trough, and the quantity of air that had entered and the gas that remained determined. Two or three and sometimes more experiments were made on each gas, until the results were found to be regular, or to vary within moderate limits. The seven gases tested were hydrogen, methane, ammonia, ethylene, carbon dioxide, sulfur dioxide, and chlorine. As an example of the data tabulated from this experiment it was found that as much hydrogen left the container in two hours as did carbon dioxide in ten hours. It was thus concluded that carbon dioxide was only one-fifth

as diffusive as hydrogen. It became evident that the diffusiveness of the gases must be inversely proportional to some function of their density—apparently the square root of their density.

Graham was next anxious to learn if, with a mixture of two gases, each gas would leave the vessel independently of the other or in the proportion of its individual diffusiveness. Not until he had done 40 experiments did he announce the results of this research. He found that the more diffusive gas left the vessel in a greater proportion than in the case of the solitary diffusion of the same gas, and the less diffusive gas in the mixture in a less proportion than in its solitary diffusion.

Graham now began to wonder about the diffusion of gases into gases other than air. To conduct this experiment he devised the following apparatus (*2*):

> ... a phial of 5.2 cubic inches, provided with a perforated cork, was filled with a mixture of ethylene and hydrogen in equal proportions. The phial was then inverted, a glass tube was thrust through the cork and likewise quickly inserted into the perforated cork of another bottle of 37 cubic inches, containing carbonic acid gas. The whole was then sunk in water until the surface of the water rose above the joinings. After ten hours the upper phial was removed and its contents washed with lime water. There remained a mixture of 12 parts, by volume, of ethylene and 3.1 parts of hydrogen.

Graham therefore decided that hydrogen must be just a little less than four times more diffusive than ethylene. If this relationship is calculated by the method often employed today, the following value is obtained:

$$\frac{\text{rate of } H_2}{\text{rate of } C_2H_4} = \sqrt{\frac{\text{mol. wt. of } C_2H_4}{\text{mol. wt. of } H_2}} = \frac{28.06}{2.02} = 3.702!$$

Only a few scientists obtained results as long ago as 1829 which agree so closely with results calculated today.

Graham's next paper, "On the law of the diffusion of gases," was read before the Royal Society of Edinburgh on December 19, 1831, and then published in the *Philosophical Magazine*, 1833 (*3*). The object of this paper was to establish with numerical exactness the following law of the diffusion of gases: "The diffusion or spontaneous intermixture of two gases in contact is effected by an interchange in position of indefinitely minute volumes of the gases, which volumes are not necessarily of equal magnitude, being, in the case of each gas, inversely proportional to the square root of the density of that gas" (*3*). If, at the beginning of an experiment, two gases are separated by a screen having minute openings, Graham discovered that the interchange of "equivalent volumes of diffusion" takes place through these openings; and if the gases are of unequal density there is a consequent accumulation on the side of the heavy gas, and loss on the side of the light gas.

Graham repeated Döbereiner's experiment with the cracked glass jar and then tried the same experiment substituting tubes, bell jars, flasks, and other glass vessels of very different forms, all of which had fissures. In every one of these vessels, filled with hydrogen, the water rose, after some hours, to a certain height. He also used Wedgwood stoneware tubes, as had been used by Priestley in some of his experiments. Neither of these two types of apparatus proved satisfactory, however, for, even when every precaution was taken, the results were not precise.

The invention of another very fundamental instrument by Graham led to the experiments which offered proof of his law of the diffusion of gases. He called this apparatus a diffusion tube which was constructed as follows (*4*):

> A glass tube open at both ends was selected, $^1/_2$ inch in diameter, and from 6 to 14 inches in length. A cylinder of wood, somewhat less in diameter, was introduced into the tube, so as to occupy the whole of it, with the exception of about $^1/_8$ inch at one extremity, which space was filled with a paste of plaster of Paris of the usual consistency for castes. After a few minutes the plaster set, and, withdrawing the wooden cylinder, the tube formed a receiver closed with an immovable plug of stucco. The less water employed in slaking the plaster of Paris the more dense is the plug, and the more suitable for the purpose. In the wet state the plug is airtight; it was therefore dried, either by exposure to the air for a day, or by placing the instrument in a temperature of 200°F. for a few hours; and thereafter was permeable by gases, even in the most humid atmosphere, if not positively wetted. The tube was finally graduated by means of mercury into hundredths of a cubic inch, and the notation, as is usual with gas receivers, counted from the top.

When a tube such as the one described above, 6 in. in length, was filled with hydrogen over mercury, the diffusion began immediately through the minute pores of the stucco and within three minutes the mercury had risen in the tube 2 in. above its level in the trough. Within 20 minutes all the hydrogen had escaped.

Graham repeated this experiment varying the mass and state of dryness of the stucco plug, the length of the tube, the period of time, the temperature, the humidity, and substituting bladder, cork, wood, or charcoal for the stucco. Seven experiments were conducted using hydrogen, and then other gases were employed. The experimental results varied but little from the theoretical results, as the following table shows:

Equivalent Diffusion in Volumes of Gases; Air = 1[a]

	By experiment	*By theory*	*Specific gravity*
H_2	3.83	3.7947	0.694[b]
CH_4	1.344	1.3414	0.555
C_2H_4	1.0191	1.0140	0.972
CO	1.0149	1.0140	0.972
N_2	1.0143	1.0140	0.972
O_2	0.9487	0.9487	0.111
H_2S	0.95	0.9204	1.1805
NO	0.82	0.8091	1.527
CO_2	0.812	0.8091	1.527
SO_2	0.68	0.6708	2.222

[a] Taken from ref. (*11*). Modern formulas are given in the first column instead of the names used by Graham.
[b] Error in printing; value should be taken as 0.0694.

The density of any gas diffused into air, if both the gas and the air are in the same state as to water vapor, can thus be obtained by the formula, $D = A/G)^2$, where G is the volume of gas submitted to diffusion and A is the volume of return air.

Graham's study of the diffusion of gases by no means ended here. Part I of his paper "On the motion of gases" (*13*) is concerned with the passage of a gas into a vacuum through a small opening in a thin metal plate. A constant volume of gas, contained in a glass jar, was first passed through a drying tube and then the time, in seconds, required for the passage of this gas through the opening in the plate was noted. Graham tried this experiment with eight gases and the final results left no doubt that "different gases pass through minute apertures into a vacuum in times which are as the square roots of their respective specific gravities; or with velocities which are inversely as the square roots of their specific gravities" (*5*).

Graham tried this same experiment with a mixture of gases next and found that in this case the velocity of diffusion was inversely proportional to the density of the mixture. Change in pressure was found to have very little effect on the rate of diffusion of air, but he discovered that "the diffusion time of air of different temperatures is proportional to the square root of its density at each temperature" (*6*).

In 1863 Graham's 16th paper on gases, "On the molecular mobility of gases" (*12*), appeared. In this paper he again discussed the diffusion of gases under pressure and for these experiments he made use of a graphite diffusiometer. This diffusiometer consisted of a plain glass tube about 22 mm. in diameter, closed at one end by a "graphite plate." In order to conduct gas to the upper surface of the graphite plate, a little chamber was formed of a short piece of glass tube, about 2 in. in length, cemented over the upper end of the diffusiometer. This short tube was closed by a cork perforated for the entrance and exit gas tubes (*7*). The diffusiometer stood over mercury and the gas constantly swept the top of the graphite plate but exerted no additional pressure, other than that of the atmosphere, because of the free escape from the exit tube. After many experiments conducted with the same apparatus, Graham succeeded in proving to himself and to the scientific world that

> . . . the permeation through a graphite plate into a vacuum and the diffusion into a gaseous atmosphere, through the same plate, are due to the same inherent mobility of the gaseous molecule. They are the exhibition of this movement in different circumstances. In interdiffusion we have two gases moving simultaneously through the passages in opposite directions, each gas under the influence of its own inherent force: while with gas on one side of the plate and a vacuum on the other side, we have a single gas moving in one direction only. The latter case may be assimilated to the former if the vacuum be supposed to represent an infinitely light gas. It will not involve any error, therefore, to speak of both movements as gaseous diffusion, the diffusion of gas into gas in one case, and the diffusion of gas into a vacuum in the other. The inherent molecular mobility may also be justly spoken of as the diffusibility or diffusive force of gases (*8*).

In 1841 Graham published "Elements of Chemistry," a textbook intended for the medical or general chemistry student. This text was considered the most valuable of the time and later editions appeared in 1856, 1858, and 1865. The first American edition was published in Philadelphia in 1843. F. Julius Otto translated it into German and later, with modifications, it was published in Germany under the editorship of Graham and Otto.

Graham's brilliant work on the diffusion of gases was first recognized in Scotland. He was the Keith Medallist of the Royal Society of Edinburgh in 1834. He was elected a Fellow of the Royal Society of London in 1836. He helped in founding the Chemical Society of London (1841) and the Cavendish Society (1846) and was the first president of each of these organizations. Besides other honors bestowed upon him by his countrymen, the French Academy of Sciences, of which he had been elected a corresponding member, awarded him the "Prix Jecker."

Graham's ideas were far ahead of those of his contemporaries. The efforts of most of his contemporaries, especially those in England, were spent on points of detail, on the filling-in of little gaps in chemical knowledge of known substances, rather than in attempts at new developments. His work, on the other hand, is noted for the fundamental nature of his investigations and for the simplicity of his procedures. The fame of Thomas Graham rests mainly on his law of the diffusion of gases and his work on the diffusion of solutions of colloids and crystalloids.

ACKNOWLEDGMENT

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RESEARCH IN THE LIBERAL ARTS COLLEGE[1]

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To many, the liberal arts college, traditionally and properly devoted to teaching, provides an uncongenial atmosphere for research. This has been stated much more emphatically. For example, former Chancellor Hutchins of the University of Chicago has said: ". . . the function of the college is to teach. It is not to conduct scientific investigation or professional training. It aims at transmitting to young people an intelligible scheme of things. This is a full-time job. It requires an excellent staff centering its attention on teaching, on improving its teaching, on making its scheme of things more intelligent and intelligible. The responsibility of adding to the world's knowledge does not rest upon the college. Its object is to communicate it" (*1*).

In indicating what I take to be the legitimate place of research in the liberal arts college, I suppose that it is proper to present some notion of the purposes of such a college. As one not directly associated with a liberal arts college, perhaps I have not the right or the competence to enter upon such a discussion. Anyway, nobody would agree with me. There has, of late, however, been much examination of conscience on the part of colleges and universities, with reconsideration of their purposes, and hence, of their curricula and techniques. This has led to such widely circulated documents as the Harvard report "On the Objectives of a General Education in a Free Society" and to many less widely disseminated but equally earnest studies on the part of numerous faculty committees. The Government has been active, too, in setting up recommendations, and even *Life* has devoted a recent issue to the problems of contemporary American education.

Although these studies were no doubt in part precipitated by World War II, they derive from our perpetual concern about education which goes back at least to Aristotle, as well as more specifically from the changing of conditions over recent decades which were brought into sharper focus by the war. For the liberal arts education, which had its traditional core in the humanities, the war, I think, only gave impetus to the long-continued assault of what has been called "operational learning"—the physical, biological, political, and social sciences—upon classical learning, and which had, long before, wrought deep changes in the liberal arts college. A great danger exists that the unwary college may give way too much before this assault, particularly since the so-called applied aspects of science have been so greatly overemphasized to the general public, which includes alumni.

What we want is balance. The humanities have been the traditional core of a liberal arts education. With this I am in agreement, provided we remember that the humanities mean the *complete* study of man in all his aspects and relationships, not excluding those with his Creator. I think it will be conceded that it is impossible and undesirable to avoid an integrated study of the sciences in any modern humane education. Sarton has said: "The history of science is the only history which can illustrate the progress of mankind. In fact, progress has no definite unquestionable meaning in other fields than the field of science. To be sure, we should not be dazzled by the Shibboleth 'progress,' for there are other features of human life which are at least as precious as scientific activities, though they are unprogressive: charity and the love of beauty, for example. But if we wish to explain the progress of mankind, then we must focus our attention on the development of science and its applications" (*2*).

For me, all this simply means that liberal education must include those studies which are precious to human life, if unprogressive in Sarton's sense; and those which illustrate the progress of man. How each college shall go about formulating its aims and how it shall translate them into educational reality are problems which each must solve. I am sure that some have taken too limited a view when they have announced their purpose to be the teaching and defense of free enterprise and the American way of life; or to serve the immediate needs of the people of their area; or, on the part of a college for women, to pledge that their graduates will be charming, happy, and safely marrried. Laudable as each of these aims may be, they seem to me to fall short of the purposes of a liberal arts college. I admit inability to satisfy myself completely about those purposes and I am sure I would never satisfy anybody else. Among many statements of liberal arts college aims which I have read, I should like to quote one which to me seems to cover many of the necessary elements. This college endeavors (*3*):

To train its students in the methods of thinking and in the use of the main tools of thought.
To acquaint them with the main fields of human interest and to direct them in the acquisition of knowledge therein.
To guide them in the integration of knowledge.
To afford them intensive training, and to encourage creative activity, within a chosen field.
To prepare them for further study or (within certain limits) for occupation after college.
To establish in them the habit of continuous scholarly growth.
To develop their power to enjoy, and to create the beautiful.
To develop their physical and mental health.
To develop their social resourcefulness.
To develop their moral and religious life.

[1] Based on talks given at Incarnate Word College, San Antonio, Texas (December 15, 1950), and before the Kentucky Association of Chemistry Teachers, University of Louisville (April 12, 1951).

To prepare them for intelligent, effective, and loyal participation in the life of family, community, nation, and the international order.

Whatever be the specific purposes of liberal arts colleges, I believe that their activities on the behalf of their students are concerned largely with intellectual achievement and that means with knowledge. I want to make it clear that knowledge does not mean a mere accumulation of facts or data, although these are necessary; it means ideas and principles as well. Now, there seem to be three aspects of knowledge. These are: the preservation of knowledge; the diffusion of knowledge, or teaching; and the discovery of knowledge, or research. There is certainly no quarrel about the major duties of the college in respect to teaching, which is its central activity. To the college also falls some responsibility for the preservation of knowledge, although the great libraries, whether in universities or elsewhere, must bear by far the greater part of the burden. The discovery of knowledge, as a rule with some brilliant exceptions, has been restricted, among educational institutions, to the universities in their graduate and professional schools. I do not believe that this should be the rule. Granted that the discovery of new knowledge, especially in the sciences, tends to be expensive and to require special training and aptitude, it is a vital aspect of learning and ways can be found for its encouragement without detriment to the prime objective of teaching, and, indeed, to its enrichment. Productive scholarship—which is but another way of saying "research"—should not be confined to universities, research institutes, and industry. And, although what I say is directed largely toward scientific research, I wish to emphasize that I have in mind research in the whole domain of learning, in the humanities and in the fields of economic and social studies.

For what reasons, then, should the college encourage research on the part of its faculty and students?

First, knowledge is the stock in trade of the college. The college, therefore, should deal with the interrelationships of all aspects of knowledge. Discovery, or research, is vital. Without it there would be no knowledge to preserve or to teach. It seems reasonable that the college should pay some attention to the means by which knowledge is acquired, not solely by the method of case histories, but also by actual participation.

Second, we need research. It is worth while in itself, as is teaching. But as never before we need new knowledge; not just specialized knowledge for an immediate practical end, but knowledge with an awareness of relationships and responsibilities; knowledge which is not just data but which goes to principles; knowledge which includes understanding. Where better can the growth of such knowledge be fostered than in college?

Third, the college faculties contain great numbers of men and women capable of doing good research. In the words of Professor Kraus, of Brown University: "There is a great reservoir of research capacity in the teaching staffs of our colleges. The lack of research production among those who teach in our colleges is regrettable. In few of our colleges is research encouraged; in some it is definitely discouraged. In some instances, the teaching load of staff members is unduly heavy; in some others, facilities for research are limited. Even so, one might expect greater research production in our colleges than is presently the case" (*4*).

Do not feel that the busy college instructor cannot find time to do good research. If the will be there, the way follows, although it is certainly not too much to ask that the college administration help smooth that way. Research productivity in the college results from a combination of able and enthusiastic instructors and a sympathetic administration which avoids over-heavy teaching loads and provides a modest budget. The research units in small colleges affiliated with the Institutum Divi Thomae have demonstrated to my satisfaction the ability of instructors in such colleges to do good research under these conditions. For example, a recent survey by Professor Sampey (*5*) indicated that one of these units ranked third in productivity of chemical research among American liberal arts colleges during the years 1942–45, and fourth during 1946–49, on the basis of publications in the *Journal of the American Chemical Society*. Some colleges are doing excellent jobs, and some active support is being given by such organizations as the Research Corporation. But much is yet to be done; the survey just mentioned lists only 37 colleges which published any research in the *Journal of the American Chemical Society* between 1942 and 1949, accounting for less than one per cent of the papers published in that journal. This showing is even worse than that revealed in an earlier survey for the years 1927 to 1941 which caused Professor Sampey "to conclude that the research published by teachers in liberal arts colleges occupies a place of negligible importance in the program of such teachers" (*6*). While the criterion employed in this survey was extremely narrow, the results are at least suggestive. All of this indicates the existence of a vast research potential among college faculties. It is shameful to waste it.

A fourth reason for research in the college involves its relationship with teaching. Participation in research vitalizes a professor's teaching. All too often the college teacher's sole contact with research occurred at the time of acquisition of an advanced degree. A revival and active use of that research training cannot help but make the teacher feel that he is contributing, if only in a small way, to the edifice of knowledge, and that he is not simply living in someone else's house, rent-free. This feeling of active participation almost inevitably enhances his "enthusiasm . . . for his subject and its dissemination," as Professor Hartman has said (*7*). I urge that research benefits teaching despite a certain basic truth in Hutchins' remark that "there is an essential conflict between teaching and research" (*8*). I do not say that all teachers should do research or that all researchers should teach; there are some who can do

the one brilliantly but not the other. But I am convinced that for most of us the one fructifies the other.

And finally, for the student, the enthusiasm which research engenders in his teacher is contagious. It can make learning, which all too often may seem static and dead, dynamic and vital. For learning and knowledge, in spite of some musty attributes, are living and moving. From this quality is born what LeGallienne somewhere aptly called "the joy of scholarship." To my mind the effect of research on the student in the liberal arts college is the most important of all. If the student can see that his teachers are taking part in a living enterprise, and best of all, if the gifted student can participate in it, he may readily be led into a habit of "continuous scholarly growth." A few students will be drawn into a life work in research; for this not only the graduate school but the world at large will be grateful. But much more important is the development in the student of this habit of continuous scholarly growth, a habit which will help him and give him joy throughout his lifetime. This is far more important than any specific bit of research which he may accomplish. For the primary concern of the college is not simply knowledge; the primary concern of the college is the student.

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VALENCE AND FORMULAS TAUGHT WITH PLAYING CARDS

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The use of devices as teaching aids in chemistry has long expedited the laborious task of making lucid the abstract or more intangible concepts. The utilization of models, figures, illustrations, and slides has become a necessary adjunct to the successful pedagogue's itinerary. Cards on which the chemical symbols were written proved valuable to such investigators as Newlands, Meyer, and Mendeleev who sorted the elements and catalogued their similarities. Recently, playing cards have been used to good advantage in teaching interrelationships of the periodic system.[1] The use of blackboard-size drawings of atoms and molecules has proved time saving, stimulating, and effective, especially in the secondary schools and in the Armed Forces training groups.

As a result of an endeavor to simplify the teaching of valence and other features of combining capacity to students of undergraduate organic chemistry, the playing card concept was used to good advantage by marking the cards with the chemical symbols (one element per card) surrounding which the correct number of valences were represented as dash marks. These

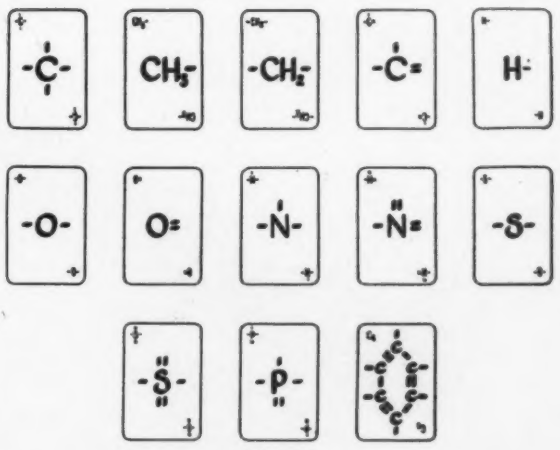

Figure 1

[1] Maxwell, J. A., J. Chem. Educ., 27, 510 (1950).

were made up according to the ratios of statistical occurrence of the most abundant elements in organic compounds (*i. e.*, C, H, O, N, S, and P).

The game of "Valence" is played using a deck of 50 to 100 cards which is dealt in hands to the participating students. These hands are then played by building molecules according to the simple rules of combining capacities, which are in many cases simpler to remember than the playing rules of canasta, pinochle, or bridge. As a game the project becomes in many instances significantly more rewarding, informative, and even amusing to the student as compared to the rigors of conventional methods of teaching such concepts. Figure 1 illustrates the manner of marking the cards. It will be noted that several "kinds" of carbon, oxygen, and nitrogen are included, showing distinctions between single and double bonded elements as well as properly restricting the manner of using some of these according to the known rules of chemical combination. Tetravalent and double bonded carbon, the methyl and methylene groups, and individual elements exhibiting variable valence are shown. The benzene nucleus shown on one card implies a group of carbons functioning as a stable unit and double bond character of a different type than in acyclic combinations. It is evident that groups of atoms or radicals such as nitro, methyl, and amino might be introduced in place of the individual elements to reduce the number of cards to a smaller but equally workable number. Figures 2, 3, 4, and 5 show a few of the possible combinations. To synthesize a molecule, cards are placed so as to attach the elements by means of the dash marks, using up the full quota of dashes on each card. Hence, the idea of constructing configurations with correct valences is readily presented. Figure 3 illustrates the manner of synthesizing graphically an aldehyde (acetaldehyde) from which the corresponding acid (acetic) is derived by the simple insertion of an oxygen. Figure 4 demonstrates the use of the benzene ring in formulating benzene and nitrobenzene; the manner of building hydrocarbon chains with unsaturations is presented in Figure 5. With students lacking a knowledge of chemistry it was observed they readily acquired the ability to make appropriate "workable" combinations of elements into carbon chains with the functional groups properly appended. They were agreeably surprised in many instances after winning a hand to find they had synthesized a substance with known chemical and physical properties and with which they were already familiar (such as the acid of vinegar and alcohols of grain or wood). To aid and stimulate interest in chemistry, large placards were made up on which common functional groups were drawn as headings to columns and the properties of each type as well as examples and uses were tabulated in the columns. Using these as models, students are able to channel their card-matching efforts toward the construction of common, well-known formulas.

Figure 2

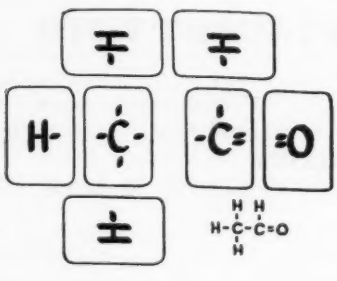

Figure 3

For chemists, colleagues, and associates who are well grounded in fundamentals of chemistry and who might have tended to look upon the playing card concepts of valence with some levity and facetiousness, a game was devised, similarly called "Valence," which has since become a favorite pastime at lunch, during train travel enroute from meetings, or at smokers and the like. This game is played by dealing seven cards to each of the participants, and follows the general principles of conquian or rummy as played with conventional playing cards. The balance of the deck is placed in the center of the table and cards are drawn and discarded in sequence by the players until a player "builds" a molecule. When a player has the proper cards with connected valence markings to lay down a molecule, he calls for a showdown (in his turn). In this particular game the scoring is done by adding the atomic weights of the atoms in the compound synthesized. The player calling the showdown may or may not have excess cards in his hands. If he does, he may win the hand only by possessing a sum of weights of unmatched cards less than that of the unmatched cards held by any other player. For instance, Player *A* may call for a showdown at his turn to draw a card from the pack, having synthesized acetaldehyde, CH_3CHO, and having one carbon and one

hydrogen left over. The sum of weights of the unmatched cards in this case is 12 (C) plus 1 (H) equaling 13. If no other player, at the time of the showdown, is able to put down unmatched cards totaling less than 13, Player *A* wins the hand with a score equal to the molecular weight of the compound he has made. Simultaneously, deductions are made from the scores of the other players equal to the sums of cards they cannot

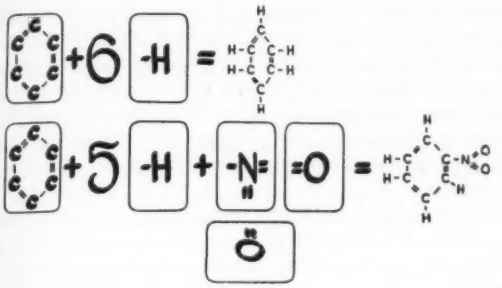

Figure 4

build into a simple known formula. Since it is nearly always possible to form combinations leading to H-O-H, NH_3, CS_2, CH_4, CO_2, and other simple molecules, many interesting possibilities result. Several game rules have evolved, such as: (*a*) the winning hand is that which contains the molecule with the highest molecular weight; (*b*) the player has an option to pick cards without discarding if he wishes to risk building a chain of atoms which requires more atoms than he is holding; (*c*) required combinations of atoms must conform to the rules of chemical combination (two hydroxyls normally do not coexist on a carbon, ketones are more stable than enols, and so forth); (*d*) molecules such as CO_2, CS_2, H_2O, and H_2S are restricted to the use of the appropriately marked type of oxygen or sulfur; and (*e*) players are prohibited from picking up more than one card at a time (from the top of discards or the top of the deck).

In order to introduce the possibility of greater return for greater risk, two cards were made up containing the benzene nucleus (C_6). The holder of such a card has the option of holding it and drawing six hydrogens to make benzene, or drawing some such acceptable number of cards to make combinations such as phenol, cresol, benzoic acid, aniline, and so forth. If successful, this card represents a 72 (the sum of the weights of six carbons) bonus. The other alternative is not to hold this card and avoid the risk of a 72-point deduction in case of a loss.

Another instructive and enjoyable pastime has been the playing of the cards similar to the game of dominoes. In this, the cards are divided among the participants and are played in sequence, face up and one at a time. The group works towards synthesizing one molecule in the center of the board but the winner of each game is the first player to complete any known formula. The molecular weight of this substance may be credited to a point score if desired.

When one considers the infinite number of games possible by matching the cards in the conventional type of deck, it is at once obvious that numerous analogous games may be improvised using chemical playing cards. Many such variations have been tried, but complete and workable rules for most are still being evolved. Practically each time a hand is played an enterprising player has some suggestion to make which enhances the instructive, or amusement, value of the particular trial of the moment. In this regard many such constructively critical comments have served to put the present system on a facile basis. The cards, used instructionally, can definitely aid students as low as high-school level to grasp quickly the concepts of combining capacity, formulas, molecular weights, and the manner of writing graphical formulas; whereas to the chemist the cards are a source of diversion and entertainment.

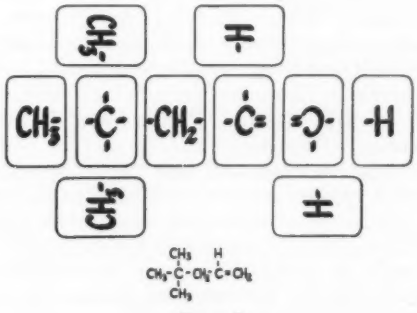

Figure 5

ACKNOWLEDGMENT

The author is indebted with thanks to Mr. Thomas Courtney Lee of the School of Medicine for preparing the illustrations.

ERRATUM

AN ERROR has been pointed out in Figure 2 in the paper, "The polarographic diffusion current and the height of the mercury reservoir," on page 495 in the September issue. A 2-volt accumulator cell should have been indicated in the top line of the circuit diagram.

STOPCOCK LUBRICANTS

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ALTHOUGH most laboratories prefer to buy one of the prepared stopcock lubricants for their general use, the occasion sometimes arises when a special lubricant is required. In addition, the preparation of these lubricants provides a useful and interesting laboratory exercise for elementary and intermediate students.

Because of their low vapor pressure and insolubility in aqueous reagents, the petrolatum-rubber paraffin lubricants are among the most popular. Shepherd and Ledig (*14*) investigated this type quite thoroughly and recommended the following formulas:

	High vacuum	*General*	*Light*
Pale crepe rubber	31 parts	6 parts	10 parts
White vaseline	24 parts	7 parts	18 parts
Paraffin, m. p. 36°C.	5 parts	1 part	1 part

The mixture of the respective ingredients is heated to 155°C. for 190 hours with continuous stirring, poured into small ointment cans, and chilled on ice immediately. After aging ten days, the grease is ready for use.

A similar recipe is given by Ardagh and Davidson (*1*). Melt 77.5 per cent of white petrolatum and 15 per cent of white paraffin (m. p. 53°C.) in an oil bath at 130°C. Add gradually 7.5 per cent of pale rubber crepe cut into small shreds. Stir continuously for six hours. While still just fluid, pour on a chilled aluminum plate. The grease is scraped from the plate and boxed. It is said to be insoluble in 15 per cent sodium hydroxide.

Still another variation will be found in Scott (*13*). Soft grease: Thoroughly mix, by melting and stirring three parts vaseline and one part beeswax. Hard grease: Add one part (*i. e.*, 1:4) of soft black rubber in small bits to the above mixture, heat to 140–150°C., and stir constantly until thoroughly incorporated.

In order to avoid the lengthy heating and stirring times, Sveshnikov (*16*) suggests mixing 20 per cent of rubber cement with 80 per cent of petrolatum and heating the mixture until the benzene has evaporated.

Although not a member of this group except for its insolubility in water, black steam cylinder oil has been suggested as an expedient by Freund (*4*).

Another group of lubricants are those using a soap base. In general these are heavy greases suitable for desiccator lids and glass joint seals. Case (*3*) recommends the following recipe: Two grams of dry soap are added to 100 ml. of a medium grade lubricating oil. The mixture is heated to 150–200°C. with stirring, for a few minutes, until solution is complete. Twenty grams of paraffin wax are then added with stirring. The cooled mixture forms a stiff jelly which retains its consistency at room temperature. Krause (*7*) recommends linseed soap melted with soap flakes. The resulting lubricant is said to be insoluble in gasoline, kerosene, benzene, and ethyl alcohol. Hodgman (*6*) suggests heating 200 g. Standard Viscous Oil No. 32 and 50 g. of aluminum stearate to about 150°C. On cooling this makes an extremely sticky, ropy, tough adhesive for ground glass surfaces. Morgan and Lowe (*9*) have patented a mixture of stearamide and glyceryl monoricinoleate which is said to be insoluble in both organic solvents and water.

In searching for hydrocarbon insoluble lubricants, a number of workers have investigated various sugar gels. Meloche and Fredrick (*8*) give as a recipe for such a lubricant: mix thoroughly 25 g. of anhydrous glycerin, 7 g. of dextrin, and 3.5 g. of c. p. *d*-mannitol until a thick paste is obtained. Heat carefully with constant stirring until the solid material is dissolved and the solution just begins to boil. Cool with occasional stirring to room temperature and store in ointment jars. To increase the viscosity add more dextrin. To increase the fluidity add more glycerin. To impart the property of a greasy paste add more mannitol. The material is resistant to hydrocarbons and chlorohydrocarbons but should not be used with water, alcohols, alphatic acids, amines, or nitrogen-containing heterocyclic compounds.

A simpler grease is described by Herrington and Starr (*5*). Suspend 9 g. of soluble starch in 22 g. of glycerol and heat to 140°C. After standing a short time decant the clear solution from the sediment and allow to cool. After twenty-four hours the mixture has the consistency of a heavy grease and is resistant to both ethyl and petroleum ethers. It is slowly souble in water but can be used in contact with water if renewed frequently.

Neither of the above two greases is satisfactory for high vacuum work. Puddington (*11*) gives the following formula for high vacuum grease. Paste carefully 1 to 3 per cent of medium viscosity polyvinyl alcohol and 15 to 20 per cent of mannitol in glycerol. Heat to 130° C. and stir until dispersion is uniform and complete. Cool with occasional stirring and keep in a desiccator. The mannitol can be replaced with about 40 per cent of sucrose and the glycerol with triethanol amine. This lubricant cannot be used with the lower alcohols and ketones.

It would seem that resins would make very desirable stopcock lubricants but very little has been reported on their use. Sager (*12*) recommends the formula: one mol of citric acid and 1.5 mols of tetraethylene glycol

are heated together at 180–185°C. for 90 minutes. The flask is removed from the oil bath and allowed to cool to room temperature. Prolonged heating of this mixture results in an infusible product. The resin obtained is clear, amber-colored, of balsam consistency, and has a marked adhesiveness. It is readily soluble in water, alcohol, and acetone but insoluble in petroleum ether and toluene. It is chemically inert and may be kept indefinitely without changes occurring in its properties.

Pearlson (*10*) modifies this formula so that it can be used for a high vacuum, thus: Heat 7.5 g. of cellulose acetate, cut in small pieces, in 45 g. of tetraethylene glycol for four hours at 140°C. with frequent stirring. Heat 30 g. of citric acid in an oil bath to 190°C. and add the cellulose acetate solution. Continue heating at 180–190°C. for 90 minutes. To remove the dissolved water, pour the solution immediately into a previously heated glass jar in a desiccator and evacuate the desiccator as rapidly as foaming permits. The resin is kept in a tightly closed jar.

All the lubricants described thus far are organic. Occasionally an inorganic stopcock grease is needed. Stephens (*15*) prepared such a lubricant by mixing 18 g. metaphosphoric acid with 35 g. of 85 per cent orthophosphoric acid. A few drops of nitric acid is added to destroy reducing substances and the mixture is heated to 300°C. in a platinum crucible. The heating time should be about 15 minutes. The melt is held at 300° for another 15 minutes, cooled to 100° and poured into bottles with rubber stoppers. It is recommended that several small bottles be used because the lubricant is spoiled by the admission of moist air when the bottle is opened a few times.

Two similar recipes are described by Boughton (*2b*) which have the advantage of being nonhydroscopic. Dissolve 10 g. of metaphosphoric acid in 100 cc. of distilled water, add 2 g. of boric acid crystals and stir until dissolved. Evaporate to about 25 ml. and add 1 ml. of 85 per cent orthophosphoric acid. Stir with a thermometer until the temperature reaches 122°C. Transfer the resulting colorless, viscous sirup to rubber or glass-stoppered bottles. The second recipe: dissolve 10 g. sodium metaphosphate (prepared by heating monosodium phosphate or sodium ammonium phosphate in platinum over a blast lamp until a quiet melt of redness is obtained) in 50 ml. of distilled water. Add 2 g. of borax and boil the mixture until the temperature reaches 106°C. Both lubricants can be used with organic liquids and vapors, acidic and neutral gases. Their use with alkaline gases or water is not recommended. They have the added advantage of being usable at elevated temperatures.

The final recipes are mixtures of inert solids with inert liquids. Boughton (*2c*) studied a number of such mixtures. Ten grams of kaolin, bentonite, graphite, or silica is mixed with 6–8 ml. of a 50 per cent saturated solution of calcium or zinc chloride. The resulting pastes have a limited use since they tend to clog openings and squeeze out. Their principal merit is their utility at sub-zero temperatures.

Hodgman (*6*) offers a modification of the above recipe which gives it better lubricating characteristics. He suggests mixing bentonite clay with glycerin until a paste of the desired consistency is obtained. This mixture is resistant to nonpolar compounds but cannot be used with polar liquids such as water or alcohol.

Boughton (*2a*) also describes an inorganic lubricant which is inert to practically every solvent. The stopcock is thoroughly cleaned and silvered by immersion in ammoniacal silver nitrate and Rochelle salt several times. An amalgam is prepared by mixing 5 g. of solder, 5 g. of Woods metal, 2.5 kg. of zinc, and 80 g. of mercury. A small amount of the amalgam is placed on the concave surface; the stopcock is inserted and gently rotated until a seal is made. The seal is permanent for several days and can be renewed by the use of more amalgam.

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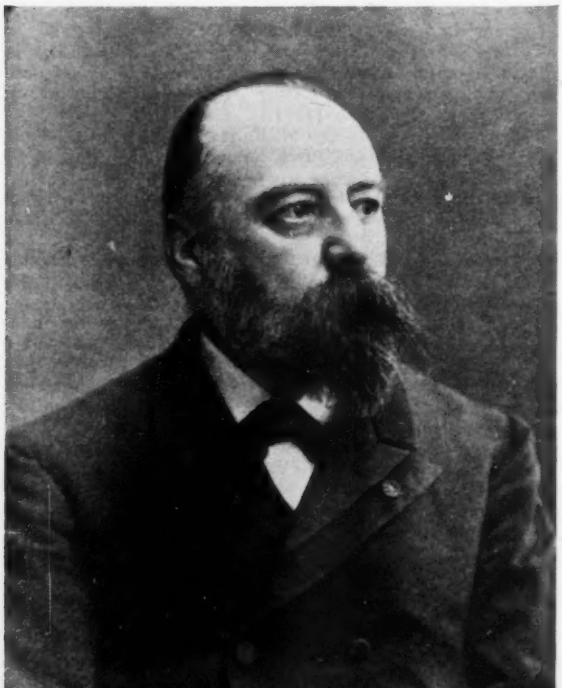
WALTER SPRING, AN EARLY PHYSICAL CHEMIST[1]

FABIAN LIONETTI and MILTON MAGER
Boston University School of Medicine, Boston, Massachusetts

THE Belgians are known to have contributed much to the advancement of chemistry. For a nation whose population compares with that of the city of New York it is remarkable to recount the impact which many of its native-born or adopted chemists have made in the historical literature. Beginning arbitrarily about 1830, when national independence was established, a significant list of notables could include Jean B. van Mons (1765–42), Jean S. Stas (1813–91), Louis Melsens (1814–86), Freidrich A. Kekulé (1829–96) (who spent nine years at Ghent), Louis Henry (1834–1913), Ernest Solvay (1838–1922), Leon Crismer (1858–1944), Fredric Swarts (1866–1940), Albert Sauveur (1863–1939), Leo H. Baekeland (1863–1944), Jean Timmermans (1882–?), and Walter Spring (1848–1911).

[1] Presented at the 117th Meeting of the American Chemical Society, Boston, April, 1950.



Courtesy of Dr. Jean Timmermans, University of Brussels

Walter Spring

Spring was a nineteenth century chemist of great originality and imaginative researches. He was born in 1848 (March 6), the son of a professor of physiology of the medical faculty at Liége. His early studies were characterized by failures in classical languages and mediocre ability in letters. This inability to succeed in his education and the resultant domestic strife led him to leave his home and family and to take a position as a laborer in an applied metallurgical laboratory (a metalsmithy).[2] It was here he developed a dexterity for manipulative skills which aided greatly in his future brilliant researches. His choice of a career in chemistry was greatly influenced by Jean Stas, a family friend, and one who was perhaps the most eminent of Belgian men of science. Guided by the inspiration of Stas, who also "sired" such other Belgian chemists as Ernest Solvay, Leon Crismer, and Louis Henry, he accomplished a successful six-year period of study in science at Liége (1867–72), which culminated very successfully in his acquiring the Ph.D. degree in the final year. The following two years were spent at Bonn where Kekulé and Clausius exercised profound influence on his aspirations and scientific ideals. In 1875 he returned to Liége where he was appointed teacher of theoretical physics. The following year he was named professor of organic chemistry; and in 1884 he became permanent examiner of the Military School of Belgium and also gained election to the Belgian Academy. In his early career he coincidentally held the position of engineer in the Belgian Bureau of Mines.[3]

Spring's research endeavors were characterized by the originality of his theoretical views and the minute exactitude of his experiments; his early work reflected a strong influence of prominent men of his time—Stas, Kekulé, and Clausius. His first lectures discussed the thesis of Stas on the role of hypothesis in science and proclaimed the important part which imagination has in research. In his first work one meets the influence of Kekulé wherein he applied his views to the interpretation of the formula of polythionic acids and that of Clausius when he undertook the study of electrostatic phenomena in mercury-filled capillary tubes. In 1880 he opened to students the first laboratory of chemistry at Liége, but his untiring devotion to pure research, his exacting standards, and his inability to adjust himself socially led finally to clashes with the administration of

[2] TIMMERMANS, J., "Histoire de la chimie," Les Presses Universitaires de Bruxelles, Brussels, 1947, pp. 99–104.

[3] POGGENDORF, J. C., "Biographisch-Literarisches Handworterbuch," Vierter Band, Leipzig, 1904, p. 1423.

the university and misunderstandings with his colleagues at the École des Mines. The result was, for Spring, a life of solitary seclusion in his laboratory.

Spring's numerous and varied investigations showed him to be quite versatile and to have interests very broad in scope. He displayed a prolonged interest in the chemistry of organic and inorganic sulfur compounds, which included preparative studies on thioacetone, hyposulfurous acid, and metal sulfides. He studied the expansion of alloys with temperature and exceptions to the law of Dulong and Petit. He is remembered mainly, however, for the studies of the effects of pressure on liquid and solid systems and for extensive researches in colloids. In his work on the compression of solids he was able to obtain pressures of 20,000 kg. per sq. cm. and demonstrated that carefully cleaned and scoured metal powder was welded into compact blocks under pressures of this magnitude. He also demonstrated that polished blocks of copper and zinc when placed in close contact and compressed produced an intermediary layer of brass, which proved the reality of diffusion of metals in the solid state. When objections were made that the heat of compression might have raised the temperature and caused liquefaction—hence favoring the reaction of the two metals —Spring refuted these by compressing phorone (melting point 28°C. under a pressure of 1 atm.) and showed the compression produced a compact block without liquefaction since a lead pellet placed in the phorone was found in the same position at the end of the experiment. He determined densities of fused solids and showed an elevation by compression which tended to a definite limit "by the abolition of pores," whereas salts of lead, zinc, aluminum, and ammonium sulfate showed an increase in specific volume with compression. The latter phenomenon was interpreted by Spring as a proof that the fluid state is a partial liquefaction involving an expansion. On the contrary, bismuth, whose specific volume diminished with fusion, contracted also when subjected to high pressure. Finally, he investigated the properties of salt hydrates and double salts and, guided by the principle of Le Chatelier, demonstrated that the volumes of these salts were greater than the sums of the volumes of the constituents, and hence decomposed under pressure. Some of his researches on the compression of powdered solids were published in Silliman's journal (*American Journal*, **36,** 1888), the first truly scientific journal in the United States. An interesting application of these high-pressure methods was the discovery that peat could be transformed into lignite and that pressure applied to layers of clay between which organic deposits were introduced produced schist rocks.

There followed an extensive array of papers on the physical properties of water, its color, purity, and solvent action which led to numerous interesting findings and an investigation of the waters of the Meuse River was conducted in which collaborative analyses were done with Proust and Roland. Repeated analyses every day for a year were done in order to verify the composition of the water and determine the variation with time. Studies on the color of the waters of the river led eventually to the preparation of optically pure water which Spring was among the first to have prepared. Water devoid of Tyndall effects was obtained by carrying down the sedimented particles with the aid of colloidal precipitated zinc and aluminum hydroxide formed by the action of ammonia on soluble salts of zinc and aluminum. In order to determine the color of pure water he observed layers as thick as 26 meters, which experimentation necessitated optical instruments of extreme precision and required skilful manipulation. Spring determined that pure water is blue whereas running water is green or brown by virtue of particles of humic acid suspended in it. The color researches were extended to organic liquids, of which hydrocarbons were shown to have a yellow color and alcohols, interestingly enough, to be green—the color intermediate between water and the paraffins. Spring did much to help prove that gravitational, electrical, magnetic, and surface tension forces between particles were not the causes of Brownian motion,[4] a controversial subject in 1900 which was being debated vigorously among his contemporary investigators. On salt solutions it was observed that true solutions formed by the alkali metals salts were optically clear, whereas with salts of heavy metals hydrolysis produced hydrates of colloidal, light-scattering dimensions. In order to make these observations he devised and constructed an optical instrument which later became the ultramicroscope of Siedentopf and Zsigmondy. Finally, Spring discovered variations in properties such as color and conductivity in solutions of electrolytes, *e. g.*, potassium bichromate, which varied with time. He interpreted these phenomena to be the result of a slow disaggregation of complexes formed by chromic acid in solutions. Numerous other labors can be listed among such varied fields as crystallography, pharmacy, organic chemistry, physics, and geography, including the publication of joint papers with Ostwald and van't Hoff.

The published articles by Walter Spring number well over one hundred and include considerations of natural phenomena such as the color of the sky, a biography of Jean Stas, and descriptive writing on the scenic beauties of the Mosan Valley.[5] Biographical literature on Walter Spring is suggestive of an eccentricity characteristic of the type who, by secluded study, lives exclusively for his professed work and renounces all social contacts. Dr. Jean Timmermans (rather anomalously) says of Spring that he was a veritable misanthrope possessed of a warm heart. Nevertheless, his proclivity for broad, imaginative research in basic science and his dextrous ability in manipulative procedures have earned for Walter Spring the distinction of having been one of Belgium's most productive and original chemists.

[4] Thomas, A. W., "Colloid Science," McGraw-Hill Book Co., Inc., New York, 1934.

[5] Crismer, L., "Oeuvres completes de Spring," Biographie, Bruxelles, 1914.

Proceedings of the

PACIFIC SOUTHWEST ASSOCIATION OF CHEMISTRY TEACHERS

OFFICIAL BUSINESS

ASSOCIATION MEETING

The Pacific Southwest Association of Chemistry Teachers sponsored an afternoon program at the annual meeting of the Pacific Division of the American Association for the Advancement of Science held the week of June 18–23 at the University of Southern California. L. Reed Brantley, President of the PSACT, presided and members from both the Northern and the Southern California Sections of the Association attended.

The first speaker, Geoffrey Keighley, Research Fellow, California Institute of Technology, talked on "How to get started in radioactive tracer research." The lecture was illustrated with actual assemblies of the minimum equipment necessary for good measurements and some examples of simple techniques in mounting samples for counting.

Willian H. Jones, U. S. Testing Company, gave a "Review of textiles, their chemical treatment and evaluation." Mr. Jones discussed the chemical treatment of natural and synthetic fibers and outlined the minimum needs for equipping a laboratory for textile testing instruction. He stressed the need for such a laboratory for the west coast area.

The final session was a stimulating panel discussion on "How profitable is undergraduate research?" Chairman of the panel was Frank L. Lambert, Occidental College, and the participating members were Norman Kharasch, U.S.C., Robert Winans, Occidental College and William V. Mayer, U.S.C. Dr. Kharasch and Dr. Winans upheld the positive side of the question, and stressed the advantages to the student of an actual experience in carrying out an original piece of independent research. Dr. Kharasch felt that only the really exceptional student should be allowed this privilege. Dr. Winans stated that such a program had been successfully carried out for a number of years at Occidental College by seniors with adequate background and demonstrated ability. On the other hand, Dr. Mayer felt that research consumed valuable time in the undergraduate schedule which could and should be used in broadening the students' basic education. He claimed that it was much easier to train a student in a specialized graduate field who already had a thorough background in the liberal arts and that such a student made a better research scientist.

SECTIONAL MEETINGS

The Northern California Section of the PSACT held its Spring Meeting at the San Mateo Junior College. The program consisted of a stimulating panel discussion on "The problems of the transfer student." J. Cason, University of California, represented the viewpoint of the large university in his discussion of the causes of discouragement and failure of some transfer students in chemistry. G. W. Cavin, San Jose State College, explained the new plan for coordinating the work of the junior colleges in chemistry with the course of study at the University of California, while H. Benninghoff, City College of San Francisco, touched on the broader aspects of the college responsibility and problems.

The following officers were elected for the year 1951-52 for the Northern California Section:

Chairman: Harry Redeker, San Mateo Junior College.
Chairman-Elect: Howard Benninghoff, City College of San Francisco.
Secretary: Florence Haimes, San Francisco State College.
Treasurer: S. W. Morse, San Francisco State College.

The Southern California Section of the PSACT combined its first spring meeting with an interesting field trip. On March 10, 1951, the group met in an auditorium in the Los Angeles County Hospital for an illustrated lecture on "Plasma proteins in disease" by Dr. John Mehl, Chairman of the Biochemistry Department in the School of Medicine of the University of Southern California. Following the lecture, Dr. Arnold Ware, Head Chemist at the Los Angeles County Hospital, explained the type of tremendous volume of work done each day in the hospital's chemistry laboratories. Dr. Ware and his assistants then took the PSACT members and guests through the laboratories, one of which contained an electrophoresis apparatus built by Dr. Mehl for research on changes of plasma proteins under pathological conditions.

On April 28, 1951, the Southern California Section of the PSACT participated in the Pacific Southwest Regional Meeting of the American Chemical Society by arranging and sponsoring one of the three sections of the program. Mr. Robert Harper, Chairman of the Southern California Section, Los Angeles City College, presided at the morning session and Dr. Harper W.

Frantz, Treasurer, Pasadena City College, presided at the afternoon session of the educational division program, which was planned by Mr. Philip Gill, Chairman-Elect, Glendale City College. The program included the following series of papers:

"The rare earth elements" by Bernard Love, Research Chemicals, Inc.
"Microcards for the chemistry library" by Sister Agnes Ann, Immaculate Heart College.
"Microanalysis: its powers and limitations" by A. J. Haagen-Smit, California Institute of Technology.
"Modern theories of adhesion and its measurements" by L. Reed Brantley, Occidental College.
"Recent advances in water analysis" by Eugene M. Diskant, Los Angeles Department of Water and Power.

The last meeting of the school year for the Southern California Section of the PSACT was held Friday evening, May 25, 1951, at Occidental College. The guest speaker, Elbert Davis, President of Plas-Kem Corporation, gave an illustrated lecture on "Recent advances in vinyl plastics." In the demonstration, Mr. Davis made and molded plastics and displayed over a hundred objects illustrating the commercial uses of typical plastics.

The officers, elected by a mail ballot for the coming year of 1951–52, were installed. Taking office were:

Chairman: Philip Gill, Glendale City College.
Chairman-Elect: Frank L. Lambert, Occidental College.
Secretary: Sister Agnes Ann, Immaculate Heart College.
Treasurer: Harper W. Frantz, Pasadena City College.

NEW MEMBERS

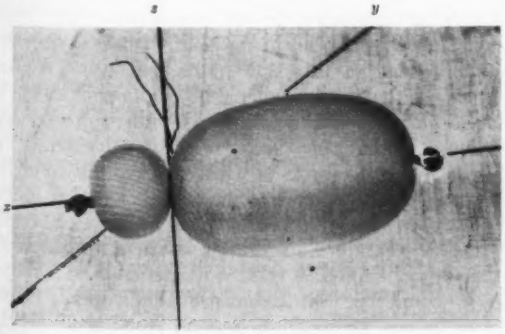
Since the publication of the membership last year[1] the following names have been added to the roll:

Northern California Section (March 15th report)
Mother Helen Hammack, Sacred Heart, Menlo Park
Mildred Johnson, San Francisco College for Women
Mother Janet Kubitschek, Sacred Heart, Menlo Park
Norman L. Lofgren, Chico State College
Sister M. Monica, College of Notre Dame, Belmont
Harry S. Mosher, Stanford University
Lowell H. Rankin, San Mateo Junior College
Ned Reed, San Francisco City College
Mother Agnes Schmit, San Francisco College for Women
J. Rae Schwenk, Sacramento Junior College
D. L. Shaw, San Francisco City College
Adelaide C. Sylva, Lux College, San Francisco
Sister M. Theresa, College of Notre Dame, Belmont

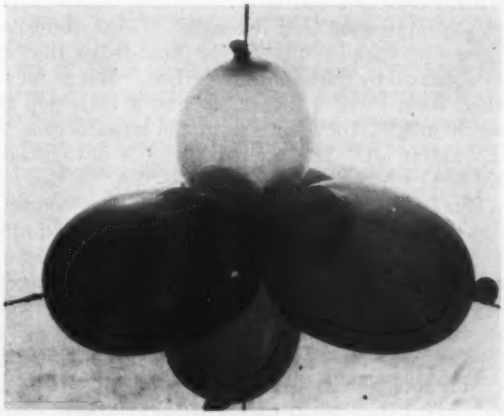
Southern California Section (June 15th report)
Imo P. Baughman, Los Angeles City College
Leslie E. Blackman, Chapman College
Dorothy Craig, Marlborough School for Girls
Sister M. Eugenia, Immaculate Heart College
Robert R. Gates, Fullerton Junior College
Hattie Hoenshall, Bakersfield College
Lowell B. Hulsebus, Bakersfield College
Lloyd N. Magnusson, Excelsior High School
Don E. Rees, California Polytechnic, San Dimas
H. R. Sheldon, La Sierra College, Arlington
Cornelius Steelink, Orange Coast College
John W. Sutton, Santa Barbara College, U. C.
Ernest H. Swift, California Institute of Technology
Harold W. Woodrow, University of Redlands

[1] See J. CHEM. EDUC., **27**, 576, 690 (1950).

THE accompanying photographs, sent in by Frank Z. Pollara of Siena College, Loudonville, New York, are of orbital models which he made for his class in general chemistry using ordinary rubber balloons. They were inspired by C. R. Noller's article "A physical picture of covalent bonding and resonance in organic chemistry" in the September, 1950 issue of THIS JOURNAL.



An sp^2 Atomic Orbital



The Two Molecular Orbitals in the Water Molecule

Report of the

NEW ENGLAND ASSOCIATION OF CHEMISTRY TEACHERS

ON THE HORNS OF THE SACRED COW[1]

ANTHONY STANDEN
The Interscience Encyclopedia, Brooklyn, New York

WHAT is it that we teach? Chemistry, presumably. And what is that? A science. And a science is an organized body of knowledge. Not just any organized body of knowledge (such as, for example, the law) but an organized body of knowledge obtained by the scientific method—and here I invite any chemist to give his own definition of "scientific method," for I am not going to define it, but will rest content with the fact that all definitions of the scientific method have, at any rate, a great deal in common.

But what are we teaching, the knowledge, or the method? The two are by no means the same, and we could teach the method, or we could teach the knowledge obtained by that method. These are the horns, and it should be noted that it is necessary to progress fairly far along both horns, for to teach a method without knowledge is meaningless, and to teach facts without scientific method is to make science indeed a Sacred Cow.

When I look at a course in chemistry, or a textbook, I get the impression that it consists almost exclusively of facts—of various bits of the knowledge that has been obtained by the scientific method. But as for this method itself—well I invariably find a little pep talk about it in Chapter I, or else in the introduction, but that's about all. The main content is always facts, facts, facts.

When I turn to the purposes for which a course in chemistry is taught I find a rather curious situation. A course in chemistry may be either for chemists, or else for nonchemists, for whom chemistry is to be only a part of their general education. Now, one would naturally suppose that the chemistry majors, the specialists who intend to be professional scientists and are going to use chemistry in their life work, would need very careful training in the scientific method, whereas this would not be so necessary for the liberal arts students. They are not going to be scientists, and one might suppose that they would not have much need for the scientific method. They are not going to be engineers, and they certainly will not receive much training in the engineering method, neither is it considered necessary to train them in the "legal method" (if there is such a thing) or in any of half a dozen other "methods" that one might name. This might be one's off-hand judgment, that the professional scientists would need to know the scientific method, whereas the others would not.

But it is quite otherwise. Any apologist for chemistry as part of the curriculum of liberal arts students always stresses the importance for these people of some aquaintance, even if a very nodding one, with the scientific method, whereas anyone engaged in the training of professional chemists is compelled to teach them facts in such enormous profusion that the amount of attention devoted to scientific method, as such, drops, percentage-wise at any rate, to a very low level.

One can look at the matter in another way and obtain the same result. What do chemists do after they graduate? Apart from the percentage who, after majoring in chemistry, go off into some other line of work, those who remain in the field—what do they do? Some of them go into the plant and become plant chemists, perhaps even chemical engineers. Some of them go into the sales department, and rise to be vice-presidents. Some of them go into advertising, or publicity, or other forms of work that can be done at a desk. Very many of the graduates will be found, twenty years later, not to have been inside a laboratory for twenty years. And yet they are still practicing chemistry, inasmuch as their work depends on a knowledge of chemistry. Note that it depends upon a knowledge of facts. It does not depend upon a knowledge of the scientific method, nor on any ability to practice it, and I suggest that many of these people know nothing of the scientific method except what they learned by hearsay in their early college days.

[1] Presented at the 13th Summer Conference of the New England Association of Chemistry Teachers, University of Rhode Island, August 23, 1951.

On the other hand the nonchemists learned far fewer facts in their college chemistry course, and they forget a higher percentage of these facts sooner, so that unless they are taught a little about the scientific method what on earth does remain when they are twenty years out of college?

A similar conclusion results from yet another approach. Why do we make laboratory work a part of the chemistry course? I am not asking *whether* we should make laboratory work a part of the course, because the answer to that question is an unqualified *yes*. I am asking for detailed consideration of exactly what it is that we are trying to achieve in the laboratory work.

First, let it be noted that we are not giving laboratory exercises in order that the students shall have the ability to do laboratory work. Only a small proportion of the students will continue to do laboratory work for more than a short while after they finish their training, and the insistence with which we give laboratory work to everybody shows that we must have some idea in our minds other than training *all* to be laboratory operators on the off chance that just a few of them will actually use this ability.

The easy answer to the question, "Why do we insist so much on laboratory work" is "To give some practice in the scientific method—of course." I think that this is a loose and ill-thought out answer. *Does* laboratory work actually teach scientific method, or have any close relation to it? Yes and no. It all depends on what you really mean by the scientific method. And now I find that I do have to define that method, or at least clarify its several meanings, after all. I will do it by the method of induction, taking several particular cases, seeing where they fall, and then seeing if we can arrive at a generalization about what is and what is not scientific method.

First of all, I would like to say that I do not think that determining a melting point can be called an example of the scientific method. It is an example of the use of a thermometer, of a Bunsen burner and a melting point tube, and it is a good example of the necessity of accurate observation. But that's about all. For, according to any definition, the scientific method comprises not only accurate observation, with measurement, but also the forming of hypotheses and the testing of the hypotheses by further experiment. But does the determination of a melting point involve the testing of a hypothesis? Again, the evasive answer, yes and no.

Before pursuing this elusive scientific method any further, I would like to say something about why we insist so strongly on students actually carrying out laboratory experiments. It may well be that my present train of thought will lead me to conclude that hardly any of the regular work of a teaching laboratory is scientific, but, even if this melancholy conclusion should be reached, I would like to make it clear that the case for having students work in the laboratory, manipulating the apparatus themselves, is not in any jeopardy. We have a cast-iron water-tight case for insisting on laboratory work, whether it is scientific or not.

The object of laboratory work, I suggest, is to permit students to know what we are talking about. Without actual work in the laboratory, the most amazing misunderstandings are possible. All of you teachers must have seen innumerable examples of this, but for amusement I will tell a story from my own experience. In England, Imperial Chemical Industries, Ltd., for whom I worked, had the idea that the bright young men, nonchemists, in the London office, could profit from brief trips to the manufacturing plants where the chemicals that they dealt with were actually made. In my branch of the great combine we were not allowed to take these young men into the plants, because we were bound to the utmost secrecy even within the company by what was known as the "Solvay agreement." I had to tell one of the young men how sodium cyanide was made. I explained how sodium, charcoal, and ammonia were all heated together in a pot. I don't known what kind of a pot the young man had in mind, but I'm sure he thought we put in the ammonia with a spoon. "What does cyanide look like?" he asked, and I told him it looked rather like sugar. "I see," he said, and wrote down in his note book "brown sugar." I hadn't said "brown" and I meant white sugar—but he had a knack for getting things wrong.

The young man was then handed over to a chemist from the sodium plant, to find out where the sodium came from. The chemist explained how electrolysis of sodium hydroxide gave oxygen at the anode, and both sodium and hydrogen at the cathode. The young man then asked an intelligent question: "How do you separate the sodium from the hydrogen?" But the chemist from the sodium plant had little sympathy with ignorance, which he mistook for stupidity; he pulled a very long face and said "Ah! That's covered by the Solvay agreement."

If you don't know what sodium and hydrogen look like, then it is a sensible question to ask how they are separated. And what does hydrogen look like? One can't see it. One can only see the glass apparatus, and the mercury or water that is used for confining it. If beginning students don't handle apparatus themselves, or at least see someone else do demonstration experiments with gases, they are apt to be totally confused as to what a gas is.

This particular misunderstanding, of not knowing what a gas is, or what water is like when it boils, is for elementary students. But even for more advanced students laboratory work must be carried out for similar reasons. Without doing a steam distillation, there can be an abject misunderstanding of what "steam distillation" means. Quite an advanced student might easily say, "Beilstein gives the melting point of this substance as 5.4°C., but the author of a paper I am reading says he got 5.5 as the melting point, so they can't be talking about the same thing." A very little experience with

melting point determinations below room temperature would show him how easy it is to be wrong by one-tenth of a degree. The same student might say, "One author has a gas of refractive index 1.00000132, while the other's is 1.0000073, there's hardly any difference between the two." If he had any practical aquaintance with the property in question, he would realize that the first digit is not significant, and the two gases really differ widely in refractive index.

I suggest that this is the true reason why we all feel that laboratory work is absolutely necessary. It is simply so that the students will know what chemistry is about. I can imagine a student, brought up on the Brønsted presentation of acids and bases, wanting to acidify a solution and looking around for a reagent bottle labeled H_3O^+. I am reminded of a member of the I.C.I. purchasing department, who is said to have designed a 2-lb. package by taking each of the dimensions of a rectangular 1-lb. package and doubling them—getting an 8-lb. package; and of another who ordered a gross of acid eggs. Even quite advanced students must have actual experience of the things they are talking about, or they will make the most hopeless mistakes.

But is all this science? Is determining a melting point to be distinguished from a thousand empirical determinations that everybody, scientist or nonscientist, makes every day of his life, things of the order of: "Is this ruler too long to go into this box? Try it and see."

If the test is that science depends upon making hypotheses and confirming them by experiment, then we have to be careful what we mean by hypothesis. To measure a table is *not* to make the hypothesis that it is four feet long, then that it is five feet long, and then six and so on until we get it right. If hypothesis means that kind of thing, then the term is far too broad, because almost anything is a hypothesis. We have to restrict the meaning of this term, or we will get a meaningless definition.

As a way of restricting the meaning of hypothesis so that it does not blow up in our faces, I suggest remembering that science is supposed to deal with generalities. It is not science to measure the length of *this* table, the height of *this* chair. There must be an element of generality, so that when a measurement is made, the result is a constant of nature, something that is the same for everybody, and stays put for all time. If a new compound is made, and if it is found to have a melting point of 22.2°C., this has some generality, for the same compound is always the same compound, and the melting point is a constant of nature. Therefore this is an example of the scientific method, at any rate in one sense, although this is probably the lowest of senses.

What about a determination in qualitative analysis? If we determine that *this* solution contains zinc ion, have we used the scientific method? Certainly not in the same sense as before. There is nothing general about *this* solution, it is a specific one. It is merely "solution *A*" in the course at this particular institution of learning. It is not the same for everybody and is not a constant of nature. I conclude, perhaps somewhat severely, that practical work in qualitative analysis is not an example of the scientific method. It corresponds to measuring the length of *this* table, with only the difference that the operation of measuring a table is exceedingly simple, whereas the operation of testing for zinc ion depends, for anything like a full understanding, upon a considerable amount of knowledge which was originally determined by other people by the use of genuine scientific method.

What about quantitative analysis? One of the distinguishing marks of any science is that, wherever possible, it makes quantitative determinations. I am afraid I cannot allow that this quantitative aspect is enough to make a determination in "quant" an example of scientific method. For what if a student does determine the percentage of copper in *this* piece of brass? This is singular, and has no generality at all. Quantitative analysis is more time consuming, and somewhat more difficult than qualitative, but in general it depends upon *less* knowledge of scientific principles. The phrase "dry to constant weight" implies many hours of tedious work, of a sort which is not rewarding in terms of any appreciable increased scientific understanding. What quantitative analysis does contribute is actual experience in the fundamentals of chemistry and in one of the most important components of almost all scientific work, namely accurate quantitative determinations. But although science depends upon accurate measurements, the scientific method is not accurate measurement. A house is not its foundations. One, but only one, of the components of the scientific method is accurate observation, preferably quantitative. The essence of the scientific method (that is to say, the most characteristic, the most scientific thing about it) is the framing and testing of hypotheses. Experiments in quantitative analysis only participate in the *essence* of the scientific method in that very limited way in which almost anything does. "Determine the percentage of copper in this piece of brass" is scarcely more scientific than "count the number of beans in this bag."

Where is science then? So far we have looked at quite a lot of chemistry, or at least covered a fair amount of ground in terms of chemistry courses, without meeting more than a fleeting glimpse of the scientific method.

I would like now to turn and approach this matter from a different direction, from the direction of mathematics. Mathematics is undoubtedly a science. It is also an exact science, although it is not an experimental science. In teaching mathematics, do we teach the scientific method? I think that most theoreticians of teaching would say that we do not. For the scientific method is usually thought of as the method of *increasing* the amount of scientific knowledge. But we do not teach mathematics for the purpose of teaching the stu-

dents the method of increasing mathematical knowledge: only for a few extreme specialists do we do that. We teach mathematics simply as knowledge and as science. Mathematical knowledge is increased, presumably, by scratching the head, tearing the hair, and worrying until a new idea comes. So is chemical knowledge, for that matter. And yet we cannot teach students to worry for an idea! One only learns that by actually doing it.

Let us follow the mathematician, after he has passed the head-scratching stage, and see what he does. Once he has his idea, he takes large quantities of paper and scribbles all over it. He writes down symbols for the thoughts in his mind. What he deals with is not, strictly speaking, the symbols, they are only to help him (at a pinch, he could do his work without them), but the ideas that the symbols symbolize. What he does is to produce a train of thought, and this train of thought is characterized by certainty and by *rigidity*, for at all points the conclusion follows necessarily from the premises, and only one conclusion follows. It is in view of this characteristic of mathematics that we call it a science.

Now experimental sciences differ from mathematics in this respect, that, being experimental, they can never lead to complete certainty. There is always a minute probability of error. But experimental investigations may, or may not, approach mathematics in their use of logical trains of thought, of more or less rigidity. I suggest that this characteristic, in addition to generality, gives another criterion by which we may judge whether any particular aspect of chemistry is scientific or not. Does it involve a logical train of thought? If it does, then it is already to that extent scientific. If it does not, then it fails, in this respect at least, of being scientific in the fullest sense.

Now let us look at some operations in the chemical laboratory and see whether they are scientific or not. The determination of a melting point has some degree of generality (although not very much) and it is scientific on that score, but it involves no logical train of thought worth calling such. What if we make a long investigation for determining the structure of a new organic chemical, say a new antibiotic? Here we must use a train of thought of considerable length. The investigation has some generality, and requires considerable logical thought. Suppose now that someone thinks of a new theory of organic chemistry, for example some new connection between structure and reactions. Such a theory is highly scientific, for it combines logical thought with great generality. And so work in chemistry can be at any level, from highly scientific to work in which there is scarcely more than a trace of the much-advertised scientific method.

To come back to the question, "What do students do in the laboratory?" I think it must be concluded that any laboratory course work contains precious little scientific method, (and this goes, of course, not only for chemistry but for any other experimental science as well). A brilliant research student may perhaps do a structure determination on an unknown compound, but he will be the only one in the laboratory doing anything at all scientific. The same student, later in his professional career, may perhaps develop such a thing as a new theory of resonance, or a new application of atomic structure theory to chemistry. When he does this, his work will be truly scientific, and in the fullest sense, but it may be noted that he will do this work not in the laboratory, but while sitting at his desk, with reference books at hand.

And so, what are we to do when we teach chemistry? We all agree that the scientific method is really more important than the facts, and I, at least, conclude that laboratory work doesn't help in the least in teaching it. And yet some idea of the scientific method has to be taught somehow. What are we to do about it?

Let us go back, for a moment, to the mathematician, whose science is easier because it is not an experimental science. The mathematics students are not, except for a few specialists, being taught the method of increasing mathematical knowledge. And yet they are scientists for all that. If they know any particular mathematical fact, they know also how to *prove* it. They can deduce the fact by a logical train from the starting point, which is a set of axioms or postulates. Our experimental science is different in that we are not allowed to postulate that results of experiments are this way, or that way, we have to take whatever results the experiments give. Our theories are scientific in so far as they are general, and are deducible by trains of thought, not from postulates but from experiments. The experiments are a necessary condition for an experimental science, and for this reason we get students to do experiments, but experiments do not by themselves constitute a science. Reasoning is also required. It should also be required of students. There should be required reasoning, as well as required reading. Facts must be known, particularly by the chemistry specialists, but the facts are only scientific if they are supported, via a train of reasoning, by experiments. This I suggest, is what we are to teach if we are really to teach the scientific method. We should teach the theory arrived at—we might consider, for example, the electronic theory of valence—and we should also teach the experiments on which the theory is based, and then make sure that the students really understand the logical connection between the two. Students should know why we believe in electrons, and why we believe in valence, and these "why's" are by no means so easy to answer as they appear. Students should be encouraged to fight back and try not to believe in our theories, for we only see the conclusive force of a train of argument by trying to deny it.

If we can get students who are well stocked with chemical facts, and who can also give a cogent argument for believing in the existence of atoms, or for the theory of electronic resonance, then I think we could claim to have progressed a satisfactory distance along both horns. Then our science is no longer a Sacred Cow.

MEDICINAL CHEMISTRY. VOLUME I

C. M. Suter, Editor-in-Chief. John Wiley & Sons, Inc., New York, 1951. vii 473 pp. 8 figs. 211 tables. 15.5 × 24 cm. $10.

THIS book is the first of a projected series encompassing reviews of topics in medicinal chemistry. The series came into being through the efforts and under the sponsorship of the Division of Medicinal Chemistry of the American Chemical Society. The authors and editor of the present volume are all members of industrial research organizations.

Such a publication can fill a valuable place in the literature of the field. In view of the enormous amount of published information, authoritative, critical reviews of single subjects are invaluable. To be of such value a review must meet certain tests as to its adequacy. These tests are discussed below and the volume is then judged with respect to them.

To be useful a review should give not only a brief history of the field but also the stimulus for the investigations reviewed. Second, the review should state specifically what literature was surveyed, the inclusive dates of the survey, and the topics surveyed. Third, reference to theories of the mechanism by which a drug performs its function is necessary to permit an examination of the hypotheses which have been tested by the structural changes investigated. Fourth, a critical discussion of the rationale, reproducibility, and relative merit of the tests used for preliminary evaluation of medicinal chemicals is essential. Fifth, the compounds or substances discussed should be organized by structure or some other common denominator. It would be preferable to have the tables of this organized data presented before or with their discussion for ease of understanding, but practical publishing policy seems to favor grouping the tables at the end. Finally, any generalities should be clearly stated, and it would be most helpful to have a statement on unsolved problems or shortcomings of the approach as revealed during the preparation of the review.

It is obvious that some of the above criteria are the province of the individual author and that others should fall to the editor. Standards of form, of presentation, and of indexing are certainly the responsibility of the latter, while the remainder falls on the shoulders of the individual author.

G. W. Anderson's review, "Antithyroid compounds," is excellent in most of the above respects. Extreme pains have been taken in the internal arrangement of the tables and in the discussion and presentation of the data as to method of assay and relative merit. Wherever possible, the compounds have been referred to a common standard. However, it would be desirable to have the unsolved problems, such as that of the variation of relative activity from rat to man in the thiouracil series (page 27), given a more prominent position.

R. R. Burtner's review, "Antispasmodics. Derivatives of carboxylic acids," is difficult reading in several respects. There is no statement as to the fields of usefulness of such compounds. The concluding date of the literature survey is given but the topics and literature surveyed are not specified. There is no discussion of or reference to possible modes of action of the compounds. The author states that it was not possible to arrange the compounds in tables structurally because of the differences in methods of reporting activities, but the very brief discussion of the screening tests only points out that standard drugs are omitted from many tests, making comparisons difficult. This decision of the author has led to the presentation of the compounds reviewed in tables arranged largely by original papers. The discussion of the tables is hampered by the fact that the compounds within a table are not numbered, making specific points difficult to cite. A reference to earlier reviews and to available pharmacological treatises on the subject would have been helpful to the reader.

C. J. Cavallito's review, "Antibiotics from plants," deals with a field that has not yet achieved the practical development of the subjects of the companion reviews. The subject is covered in a stimulating way, with extensive discussion of the types of compounds responsible for the actions found and of the mechanisms involved in these. In discussing agents of possible antibacterial value from plant sources the development of the antipneumococcal agent, hydroxyethylapocupreine, from the cinchona alkaloids is surprisingly not mentioned. The author might also have added a reference to McIlwain's interesting speculations on the role of antibiotics in plants (*Nature*, **153**, 300 (1944)) to his section on the subject. The material that is discussed in the review (pages 255–261) under the heading "Crude preparations and screening" is also covered in the tables and one feels that it is unnecessary to have it in both places.

T. P. Carney's review, "Benzoates and substituted benzoates as local anesthetics," deals with a classical field of medicinal chemistry without, however, giving the date of termination of his literature search or the precise method of making it. An extensive discussion of the testing methods is given without references to possible mechanisms of action. It would have been desirable to have a brief indication of the relative merits of the many test methods. The discussion of the well organized tabular material leads to the conclusion that the relation of structure to activity is still tentative. It is disappointing not to find reference to other reviews in the field.

E. J. Fellows and G. E. Ullyot have reviewed "Analgesics: aralkylamines." They have defined the field of structures which are being reviewed without giving the dates or the means by which they have been searched. Certain definite conclusions are drawn as to the relation of structure and activity of the majority of compounds covered. It is difficult to follow the discussion of the tabular material since the compounds reported in the tables are identified by arbitrary code numbers which are not in numerical order. Thus, Code No. 299 appears on the first page of Table 21 and No. 157 on its fifth page. It is a poor arrangement. Again no reference to mode of action or mechanism is made.

The concluding review is that of John Lee on "Analgesics: B. Partial structures related to morphine." This review has the virtue of being the most coherent in the book but this has been achieved by confining the work largely to results on compounds from the author's laboratory without placing this work in relation to the other extensive investigations on partial structures related to morphine. This review is more clearly presented than its companions, as the tabular material appears continuously throughout the text. However, the objectionable use of arbitrary code numbers in the tables is present. In addition, only the compounds from the author's laboratory are tabulated and it is not clear as to what part of the literature is covered. The conclusions as to the effect of structure on activity point out the lack of a satisfactory generality.

It is one of the objects of this publication to provide "correlation of structure and (biological) activity" and to present "discussions of the relationships between chemical structure and pharmacological action." Throughout the work there is caution on the part of the various authors in drawing such conclusions. It seems unfortunate, in view of this, that only two of the authors have inquired beyond the pictorial representation of the compounds studied as to what property or properties of the compounds influence the biological activity. Anderson and Cavallito discuss this phase of their topics. None of the other authors has expressed the fact that possibly something besides the structural formulae of the compounds might be responsible for their difficulties of interpretation. It would seem that it is time to recognize that structure and activity are only secondarily related; that both have a great deal to do with the properties of the compounds concerned but only indirectly do they deal with each other. That

this is not explicitly stated is disappointing in reviews of this expected quality.

The index to the volume seems satisfactory in its reference to types of compounds and in view of the detailed nature of the tables of contents of the reviews and arrangement of the tabular material.

Within the above limits the publication is a good, if in places indefinite, reference work. A good deal of the indefiniteness is the responsibility of the editor. The publication is well prepared physically and typographical errors are few.

JACKSON P. ENGLISH

American Cyanamid Company
Stamford, Connecticut

• INDUSTRIAL AND SAFETY PROBLEMS OF NUCLEAR TECHNOLOGY

Edited by *Morris H. Shamos*, Assistant Professor of Physics, and *Sidney G. Roth*, Assistant Professor of Mathematics, New York University. Harper and Brothers, New York, 1950. xiii + 368 pp. 50 figs. 12 tables. 14 × 21.5 cm. $4.

This book consists of a collection of papers presented at a Conference on Industrial and Safety Problems of Nuclear Technology held at New York University in January, 1950. As stated in the preface, "It is hoped that this volume will help to fill an urgent need by presenting in a single source certain fundamental information which is felt to be of particular importance to industry."

The book is divided into four main sections. In Part I the organization, operation, plans, and programs of the U. S. Atomic Energy Commission are discussed. Explanations of the contract and patent policies of the Commission are presented in some detail.

Part II deals with the properties and uses of radioactive materials. Both the present and the potential uses of radioisotopesi n all fields of research and industry are described and illustrated. Readers who are not familiar with the wide variety of uses of radioisotopes may find in these papers the mention of a use which would be helpful in their own research.

The problems of designing laboratories for handling radioactive materials and the techniques appropriate for conducting experiments in such laboratories are considered in Part III. Specific recommendations for laboratory design and useful data for shielding calculations, etc., are given.

Part IV deals with the hazards, safety problems, and the insurance aspects of nuclear technology. In the authoritative discussion of health protection and hazards may be found advice which should be of value particularly for users of radioisotopes who do not have direct assistance from a "Health Physics" staff within their organization. The reasons behind the recommended safety procedures are properly emphasized.

Two panel discussions which were transcribed from the Conference are included in the appendix.

Users and potential users of radioisotopes will find the answers to many of their questions in this book.

HERBERT M. CLARK

Rensselaer Polytechnic Institute
Troy, New York

• THE NEW PHYSICS: TALKS ON ASPECTS OF SCIENCE

Sir C. V. Raman. Philosophical Library, New York, 1951. 144 pp. 14 × 21 cm. $3.75.

This set of 19 lectures is written for the layman rather than for the scientist. The lectures were originally used as radio talks in India and it is evident that radio science programs in India have this in common with those in America, that they tend to be informative rather than instructive, to exhibit the ramifications of science, and to convince the layman of the importance of scientific work, rather than to show in any particular instance how the scientist works. It would appear that Professor Raman has written for an audience somewhat more mature intellectually than the American radio audience when he tells his listeners that the proposition "that every non-centro-symmetric crystal is piezo-electric or pyro-electric is not necessarily true," or refers to "Doppler displacements," or to representing a variable "in a diagram on a logarithmic scale."

The author is Professor of Physics at the University of Calcutta, discoverer of the Raman effect—for which he received the Nobel Prize in 1930—founder of the Indian Academy of Sciences, and cofounder of the Indian Journal of physics. The nature of the topics discussed is a testimony to the variety of his interests. Among the subjects of the lectures are shellfish, geometry in nature, the soil, water, weather, lightning, crystal structure, cosmic rays, and a review of G. W. Morey's book, "The Properties of Glass."

The style is lucid, nonmathematical, and about what one might expect a physicist to say in an after-dinner conversation with a group of, perhaps, lawyers, merchants, and government officals. It is admittedly a difficult task for the expert to explain his specialty to the layman and to convey an idea of why the expert is so enthusiastic about his work. Professor Raman has accomplished this aim. The price, however, seems high for a 144-page nontechnical book.

WALTER B. KEIGHTON

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Swarthmore, Pennsylvania

• TREATISE ON POWDER METALLURGY. VOLUME II: APPLIED AND PHYSICAL POWDER METALLURGY

Claus G. Goetzel, Vice-President and Director of Research, Sintercast Corporation of America, New York. Interscience Publishers, Inc., New York, 1950. xviii + 910 pp. 628 figs. 285 tables. 15.5 × 23.5 cm. $18.

According to the subtitles, Volume I [reviewed J. Chem. Educ., **26**, 623 (1949)] of this encyclopedic treatment of the field of powder metallurgy dealt with the "technology of metal powders and their products." Little information was given on the physical and mechanical properties of sintered materials. Part 1 of the present second volume of the treatise discusses the field of applied powder metallurgy and the properties of industrial materials and products; and Part 2 is concerned with physical powder metallurgy and a practical evaluation and a theoretical analysis of the materials, products, and processes. The third, and final, volume will cover the technical and patent literature of the field of powder metallurgy in the form of a classified and annotated bibliography. It is an awe-inspiring task that the author has set for himself and the warm reception accorded the first volume must have been very gratifying to him and to the publishers. Volume II comes up to the high expectations engendered by a study of Volume I.

Besides the practical aspects of powder metallurgy, Volume II presents in Chapter 35 a 62-page summary of the various ideas of atomic behavior that have been advanced to account for the phenomenon of sintering. This section will be of particular interest to physical chemists who are concerned with interatomic forces, surface tensions of solids, lattice vacancies, and plastic flow, all of which bear upon the new science of the "physics of the solid state." Instead of presenting a statement of his own beliefs, Dr. Goetzel has been content to present a composite summary of several separate attempts to explain sintering, though he states his preference for the more conservative concepts. He finds (on page 835) that "one of the chief causes for the slow development of a science of powder metallurgy lies in the fact that the experimental evidence is in many respects extremely scarce; in other instances, where abundant, the results constitute data of interest only for specific applications and often are insufficiently organized for scientific research and theoretical conclusions. . . any

satisfactory and complete theory must be capable of explaining *all* the established and uncontested facts . . . based on a scrutiny of the mass of experimental results that have been reported in the published literature over the last twenty-five years." In one of the latest theories advanced to explain sintering, the process effects a lowering of the free energy of the system by nucleation, recrystallization, and grain growth as progressive stages, and the respective rates are related to the nature of the surface of the particles. Accordingly, particle shape and particle size distribution have specific effects on the diffusion phenomena. Except for interferences caused by evolution of gases, the sintering is analogous to fusion. But it can occur at temperatures far below the normal melting point of the lowest-melting phase.

In addition to the expected surveys of the properties of refractory metals, cemented carbides, electrical materials, magnetic materials, friction products, dental alloys, and so on, considerable attention is given to an honest comparison of properties of powder metal products with those of the same materials manufactured in other ways. Reference is made as well to materials and processes that have not been adopted for either technical or economic causes. The treatise is very comprehensive, richly documented, and, so far as is humanly possible, unprejudiced. It will remain a monument to the amazing energy and competence of its author. As a result of the publication of this truly encyclopedic work, the art of powder metallurgy, as well as the science, will spread at an accelerated pace.

LAURENCE S. FOSTER

Belmont, Massachusetts

● THE AMERICAN FIAT REVIEW OF GERMAN SCIENCE (1939–1946). VOLUME 34: THEORETICAL ORGANIC CHEMISTRY

Walter Hückel, **University of Göttingen, and *F. Seel.* Translated, edited, and published in cooperation with the Office of Technical Services, Department of Commerce, U. S. Government, by The O. W. Leibiger Research Laboratories, Inc., Petersburg, N. Y. Lithoprinted by The M. H. Davidson Co., New Haven, Conn., 1951. x + 213 pp. 6 figs. 8 tables. 13.5 × 22 cm. $5.30.**

"It is unusual that a development report in a science comprises the achievements of a single country, in this case Germany. However, the special circumstances of the present time make this necessary," for none of the research workers of Germany use the terminology adopted in the Anglo-Saxon countries.

"Although other German research workers are not historically bound in a similar manner, they were not able to disregard the historical development in theoretical organic chemistry in Germany if they wished to avoid any discrepancy with other organic chemists not gaged to theory. May whoever reads this report as a non-German, therefore, have an understanding for the individuality of the representation and for the ways selected for German theoretical research in organic chemistry."

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Chapter II on Stereochemistry covers configuration, polymembered rings and bridge-ring systems, molecular structure and rate of reaction, configuration and basicity, stereoisomerism of the double bond, stereoisomerism of inner-complex salts, and optical activity.

Chapter III deals with free carbon radicals and biradicals, and Chapter IV on Reaction Processes deals with schemes for substitution and replacement reactions, addition reactions, and rearrangements. A bibliography concludes each chapter.

The book contains numerous illustrative equations and presents the background for the organic research program in Germany during the past two or three decades.

The lithoprinting and binding are both very good and this book should prove to be a valuable addition to the library of any organic chemist.

E. F. DEGERING

Buckman Laboratories, Inc.
Memphis, Tennessee

● THE AMERICAN FIAT REVIEW OF GERMAN SCIENCE (1939–1946). VOLUME 35: THEORETICAL ORGANIC CHEMISTRY, Part II

Walter Hückel, **University of Göttingen, *J. Goubeau, G. V. Schulz,* and *G. Scheibe.* Translated, edited, and published in cooperation with the Office of Technical Services, Department of Commerce, U. S. Government, by The O. W. Leibiger Research Laboratories, Inc., Petersburg, N. Y. Lithoprinted by The M. H. Davidson Co., New Haven, Conn., 1951. xii + 221 pp. 43 figs. 21 tables. 13.5 × 22 cm.**

"Military Government of British, French, and U. S. Zones of Germany by means of their respective FIATs (Field Information Agency, Technical) present this volume of the 'Fiat Review of German Science' in the hope that it will assist in informing international science of research done in Germany through the war years. It is believed that this and its companion volumes will present a complete and concise account of the investigations and advances of a fundamental scientific nature made by German scientists in the fields of biology, chemistry, mathematics, medicine, physics, and sciences of the earth during the period of May, 1939, to May, 1946.

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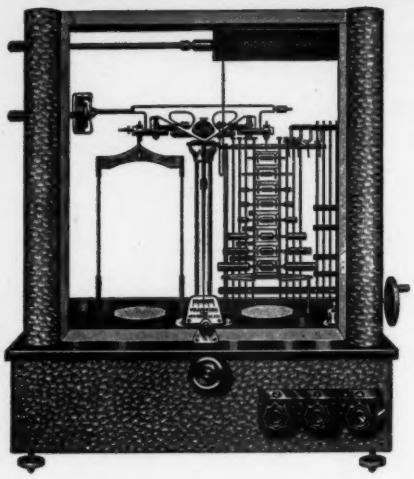

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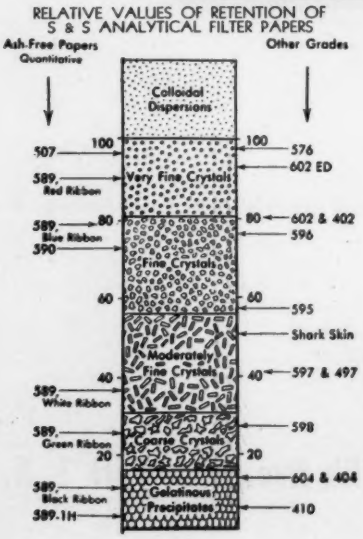

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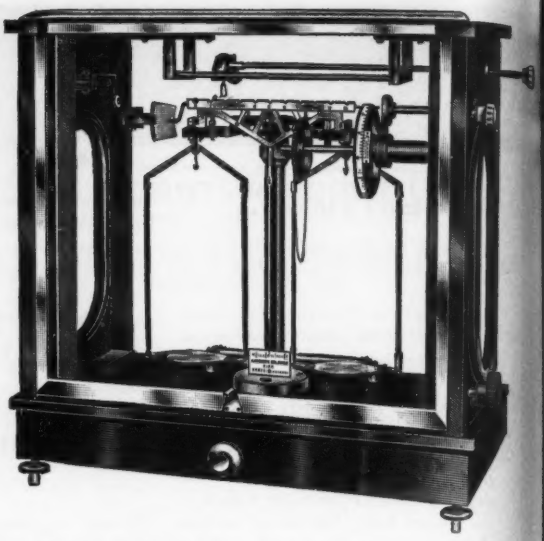

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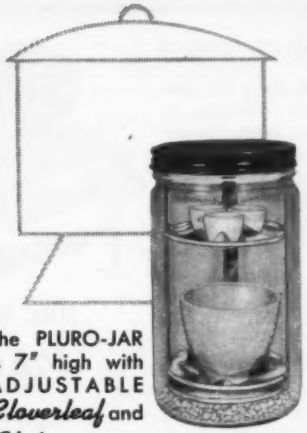

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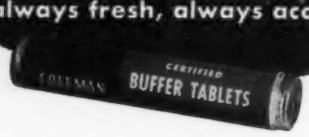

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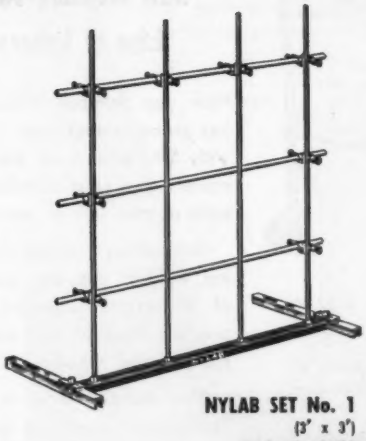

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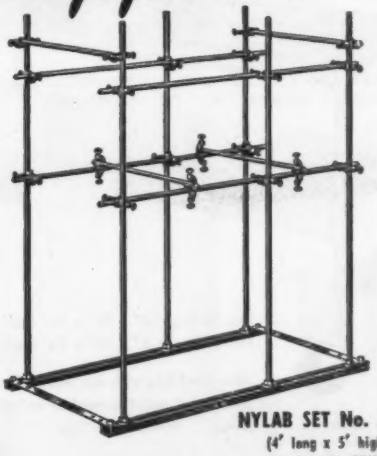

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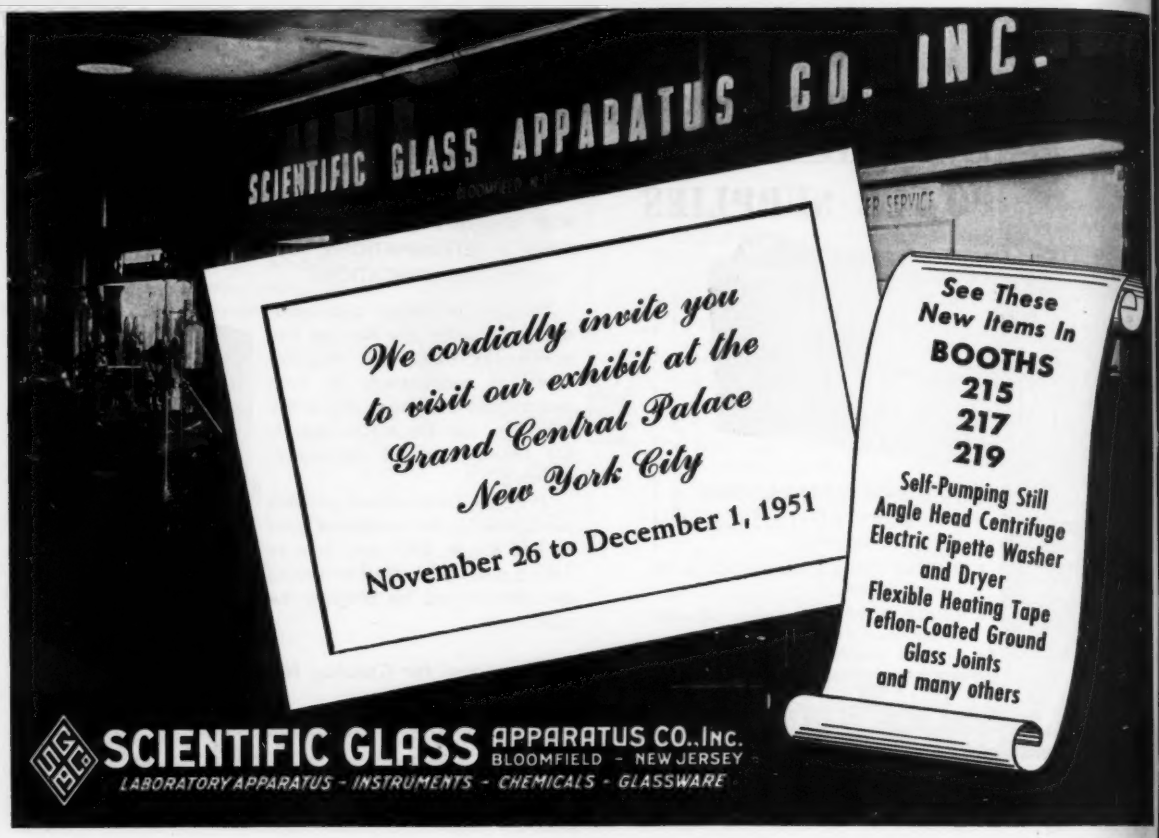

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Out of the Editor's Basket

Portable Electric Oven

A new low-priced portable electric oven with a temperature range of 100° to 800°F. has just been announced by Grieve-Hendry Co., Inc. Called the Model HT, it is recommended by the manufacturer for processing at the higher temperatures required for stress relief.

This oven features uniform temperature throughout. Construction permits the nesting of one oven on top of another. They can be used in groups or banks and can be operated individually or selected ovens in the group can be cut out or heated at different temperatures.

Construction is heavy gage steel with fibreglass insulation. It will operate from any 110-volt outlet. No special wiring is required. Thermostat, outside reading thermometer, pilot light, drip pan, and lower shelf are standard equipment. Additional removable shelves and oven stand are available.

Size is 30 in. wide × 25 in. deep × 24 in. high, outside. Inside dimensions are 22 in. wide × 18 in. deep × 16 in. high.

Full details may be had by writing the manufacturer Grieve-Hendry Company, Inc., 1101 N. Paulina St., Chicago 22, Illinois.

Electronic Relays

Three new Electronic Relays, designed to meet specific laboratory performance and price requirements have recently been introduced by the Emil Greiner Co., 20-26 N. Moore Street, New York 13, N. Y.

Model E-3, one of the new relays, is claimed to be the only one with d.-c. control, which eliminates pickup and capacitive effects, permitting the use of unlimited lengths of leads. In addition, this model has an isolating transformer which permits full freedom in grounding either side of the line or control circuit.

Lower priced are two simplified relays, Models E-2 and E-1. Similarly to Model E-3, these relays provide completely sealed contacts and means for easily changing from normally on to normally off operation.

All three units are carefully engineered for mechanical ruggedness and convenience of installation and operation. Standard size knockouts provide entry for B-X cables, and convenient screw terminal boards make electrical connections easy.

Complete descriptive bulletin is available on request.

Flexible Heating Tape

Scientific Glass Apparatus Co. Inc., Bloomfield, New Jersey, announces a new Flexible Heating Tape for small glass vessels of standard or odd shape—such as distilling or fractionating columns.

The chief advantage claimed for this tape (designed to operate at "black heat") is the fact that it produces high wattages without excessive heating of the wire. Being well below the glow point, it eliminates the danger of fire in the presence of inflammables. This is possible only because of its unique construction which

By any standard of comparison Tygon *flexible-plastic* Laboratory Tubing stands head and shoulders above the crowd. It is clear, for solution visibility; flexible, for ease in handling; tough for extra long life. It is chemically inert, and non-toxic. And available in a range of sizes to meet every laboratory need.

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No. 43 is No. 40 washed with solvent to make it fat free.

No. 44 is thin with low ash, retentive and used when minimum absorption of the filtrate is essential.

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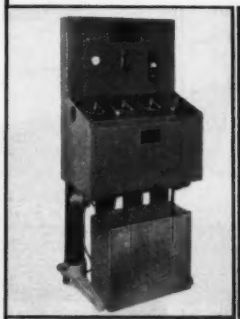
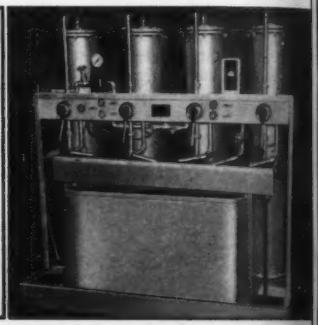
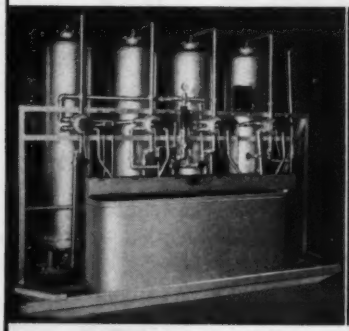


Barnstead Four-Bed Demineralizer provides pure water for hot seal tank in anodizing. 30 gal/h.



Four-bed Barnstead Demineralizer produces pure, sparkling-clear rinse water for pharmaceutical plant. 200 gal/h.



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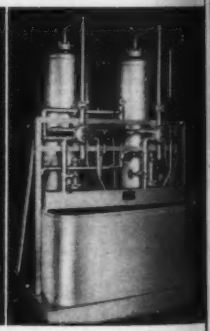

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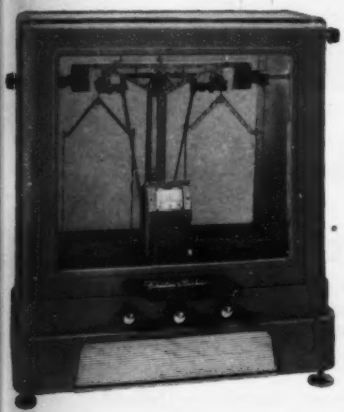
For further information write to Scientific Glass Apparatus Co., Inc., Bloomfield, N.J.

New "Projectomatic" Balance

Latest addition to the line of completely redesigned Christian Becker Balances is the "Projectomatic" model AB-1.

Although designed for fast repetitive weighing the Balance is ideally suited to regular Laboratory work. It is the only Balance of this type with double pans making taring (compensating for container weight) possible.



Projection is accomplished through a new optical system of special precision-ground lenses developed through more than 2 years of experimentation and research. The result is a clear sharp image on a screen which is scientifically positioned for ease of readability. A lamp behind the heat-insulated back panel is projected through a transparent microscale. The light is automatically switched on and off as the beam is released and arrested. A vernier on the screen provides direct and positive readings from $^1/_{10}$ mg. to 100 mg., each side of the center zero.

Automatic dampers at each end of the beam arrests oscillation quickly, further speeding a positive reading.

The new "Projectomatic" also has advantages common to all the new Christian Becker metal-cased Balances: Improved lighting and less eye strain in the tapered case, greater working access, less reflection and background distraction, improved beam arrestment that stops knife-edge dulling, greater stability through functionally engineered interlocked metal case construction of rigid long-life nonmagnetic aluminum, built-in vibration eliminators, individual pan arrests, and modern design that is easier to keep clean.

The Balance is currently in production at the Christian Becker division of the Torsion Balance Company in Clifton, New Jersey.

New Fluorescent Illuminator

A greatly improved balance illuminator has just been announced by the Meyer Scientific Supply Company, Inc.

The ballast (a special choke coil located in the housing of every fluorescent light) here is located on the wire near the plug. With this change in position, the source of magnetic interference, constant vibration, and cyclical hum are eliminated. Placed six feet away from the balance, it can do no harm. None of the heat or other emission from the ballast can interfere with the proper operation of the balance.

Finished in gray wrinkle, the "Royalton Balance Illuminator" is made with soft rubber feet and is set on top of the balance. It requires no screws, clamps, or other fittings to fit any conventional type analytical balance. All electrical components are Underwriters Laboratory Approved for operation on 115 volt 60 cycle a.c. The illuminator comes complete with G. E. lamp, ready to plug in.

Additional literature may be obtained from Meyer Scientific Supply Company, Inc., 215 North Eighth Street, Brooklyn 11, N. Y.

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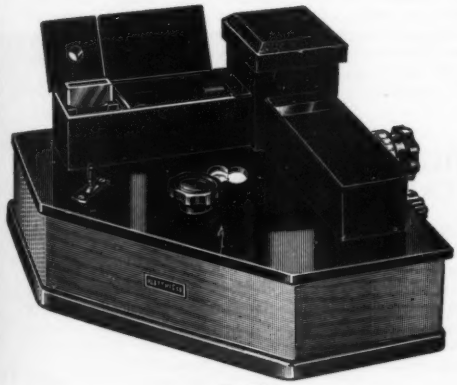

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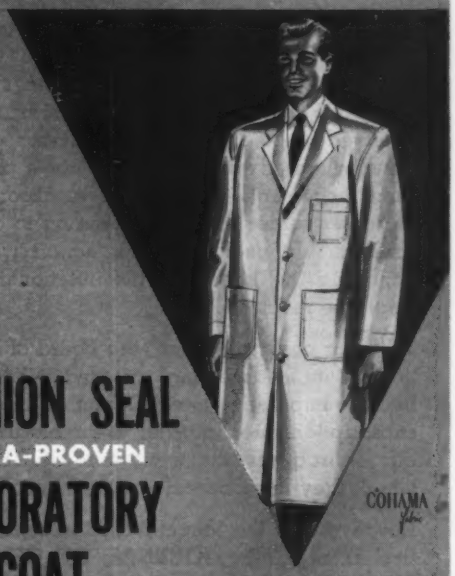

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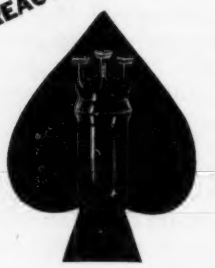

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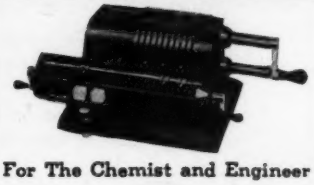

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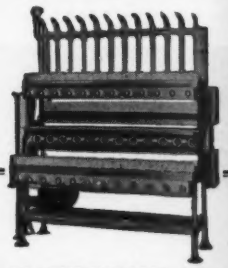

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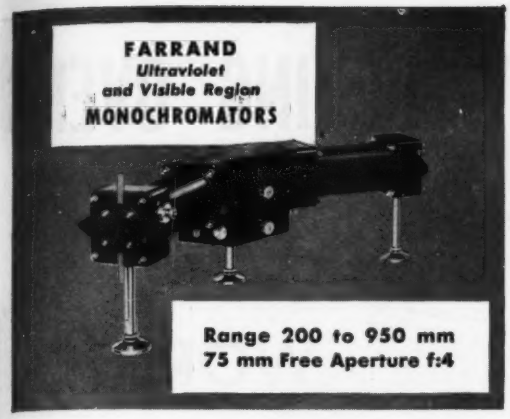

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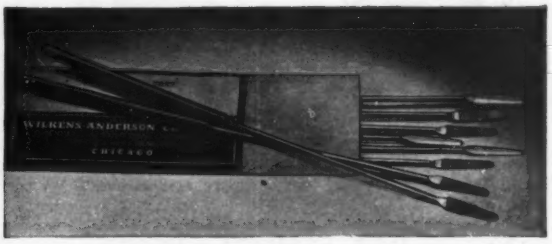

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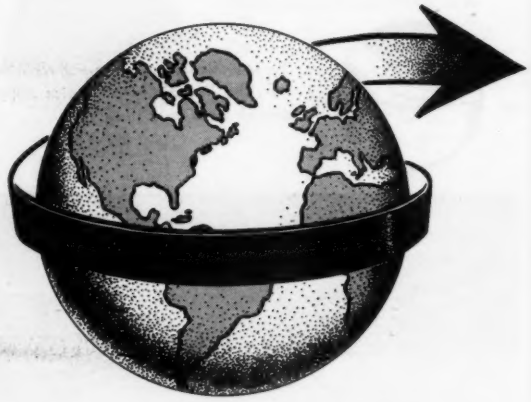

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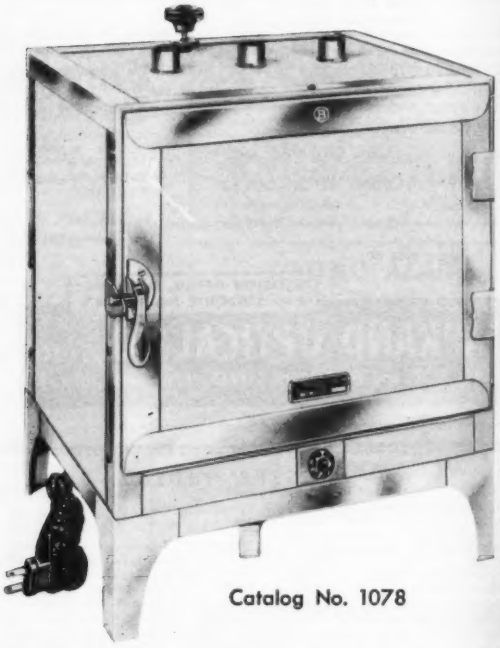

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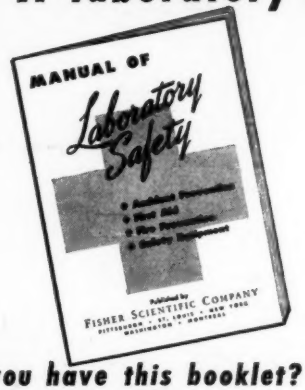

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